WEST	
Help Logout Interrupt	
Main Menu Search Form Posting Counts Show S Numbers Edit S Numbers Preferences 0	Dases
Search Results - Terms Documents 14 and 12 8	
US Patents Full-Text Database US Pre-Grant Publication Full-Text Database JPO Abstracts Database EPO Abstracts Database Derwent World Patents Index IBM Technical Disclosure Bulletins L9 Refine Search	
Recall Text Clear	
Search History	

DATE: Tuesday, August 12, 2003 Printable Copy Create Case

Set Name	Query	Hit Count	Set Name
side by side			result set
DB = USPT, PG	PB,JPAB,EPAB,DWPI; PLUR=YES; OP=ADJ		
<u>L9</u>	14 and 12	8	<u>L9</u>
<u>L8</u>	L7 not 16	5	<u>L8</u>
<u>L7</u>	12 and 13	7	<u>L7</u>
<u>L6</u>	12 and L5	2	<u>L6</u>
<u>L5</u>	13 same L4	75115	<u>L5</u>
<u>L4</u>	layer\$4 or coat\$4	3827928	<u>L4</u>
<u>L3</u>	granul\$7	366579	<u>L3</u>
<u>L2</u>	"class a" with 11	37	<u>L2</u>
<u>L1</u>	biosolid\$1 or (bio-solid\$1)	307	<u>L1</u>

END OF SEARCH HISTORY

Generate Collection

Print

Search Results - Record(s) 1 through 2 of 2 returned.

☐ 1. Document ID: US 20020162795 A1

L8: Entry 1 of 2

File: PGPB

Nov 7, 2002

PGPUB-DOCUMENT-NUMBER: 20020162795

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020162795 A1

TITLE: Methods and apparatus for biological treatment of waste waters

PUBLICATION-DATE: November 7, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Pollock, David C. Calgary CA

US-CL-CURRENT: 210/621; 210/626

Full Title Citation Front Review Classification Date Reference Sequences Attachments Claims KWIC Draw Description

1 2. Document ID: US 20020098982 A1

L8: Entry 2 of 2

File: PGPB

Jul 25, 2002

PGPUB-DOCUMENT-NUMBER: 20020098982

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020098982 A1

TITLE: Production and use of biosolid granules

PUBLICATION-DATE: July 25, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Burnham, Jeffrey C. Naples FL US

US-CL-CURRENT: 504/359; 504/117, 504/367, 71/64.02, 71/64.11

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC Draw, Desc Image

Generate Collection

Print

End of Result Set

Print Generate Collection

L8: Entry 2 of 2

File: PGPB

Jul 25, 2002

PGPUB-DOCUMENT-NUMBER: 20020098982

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020098982 A1

TITLE: Production and use of biosolid granules

PUBLICATION-DATE: July 25, 2002

INVENTOR-INFORMATION: . . . ^ NAME CITY STATE COUNTRY RULE-47

Burnham, Jeffrey C. Naples FLUS

APPL-NO: 10/ 051387 [PALM] DATE FILED: January 22, 2002

RELATED-US-APPL-DATA:

Application is a non-provisional-of-provisional application 60/262631, filed January 22, 2001,

Application is a non-provisional-of-provisional application 60/272469, filed March 2, 2001, . . .

INT-CL: [07] A01 N 25/28, A01 N 25/00, A01 N 63/00, A01 N 25/12

US-CL-PUBLISHED: 504/359; 504/117, 504/367, 71/64.02, 71/64.11 US-CL-CURRENT: 504/359; 504/117, 504/367, 71/64.02, 71/64.11

ABSTRACT:

This invention relates to the production and use of encapsulated and/or concentrically-constructed fertilizer or bioremediation granules such as, for example, granules of 0.5 mm to 10 mm in diameter constructed so that there are at least two components to the granule including a core with a surrounding capsule or a core with one or more concentric layers that are distinguishable from the core with respect to nutrient content, density, hardness, solubility, composition, microbial content and permeability, as in permeability to odors or the permeability of nutrients that might volatize to the atmosphere or leach into the soil. The basic idea was to create a method for manufacturing and using fertilizer granules, which incorporate multiple concentric layers or a core plus an encapsulating outer layer.

Generate Collection Print

L8: Entry 1 of 2

File: PGPB

Nov 7, 2002

PGPUB-DOCUMENT-NUMBER: 20020162795

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020162795 A1

TITLE: Methods and apparatus for biological treatment of waste waters

PUBLICATION-DATE: November 7, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47.
Pollock David C. Calgary CA

APPL-NO: 10/ 083995 [PALM]
DATE FILED: February 25, 2002

RELATED-US-APPL-DATA:

Application is a non-provisional-of-provisional application 60/271201, filed February 23, 2001,

INT-CL: [07] <u>C02</u> <u>F</u> <u>3</u>/<u>02</u>

US-CL-PUBLISHED: 210/621; 210/626 US-CL-CURRENT: 210/621; 210/626

REPRESENTATIVE-FIGURES: 1

ABSTRACT: In a vertical shaft bioreactor, improved devices and methods are provided for enhanced secondary and/or tertiary treatment of wastewater, including residential, municipal and industrial wastewater. The devices and methods of the invention are useful for enhanced secondary wastewater treatment, including BOD and TSS removal. Tertiary treatment can alternately or additionally be achieved in the bioreactor with nitrification of ammonia, with nitrification and denitrification, and with nitrification, denitrification, and chemical phosphorus removal. A vertical shaft bioreactor is also provided which achieves thermophilic aerobic digestion and pasteurization of sewage sludges, optionally to produce class A biosolids.

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit of U.S. Provisional Application No. 60/271,201, filed Feb. 23, 2001.

Generate Collection Print

L8: Entry 1 of 2

File: PGPB

Nov 7, 2002

DOCUMENT-IDENTIFIER: US 20020162795 A1

TITLE: Methods and apparatus for biological treatment of waste waters

Abstract Paragraph (1):

In a vertical shaft bioreactor, improved devices and methods are provided for enhanced secondary and/or tertiary treatment of wastewater, including residential, municipal and industrial wastewater. The devices and methods of the invention are useful for enhanced secondary wastewater treatment, including BOD and TSS removal. Tertiary treatment can alternately or additionally be achieved in the bioreactor with nitrification of ammonia, with nitrification and denitrification, and with nitrification, denitrification, and chemical phosphorus removal. A vertical shaft bioreactor is also provided which achieves thermophilic aerobic digestion and pasteurization of sewage sludges, optionally to produce class A biosolids.

Summary of Invention Paragraph (31):

[0030] Despite the foregoing developments and advancements in wastewater treatment technologies, there remains an urgent need in the art for improved wastewater treatment systems that can satisfy a broadened range of uses and perform expanded and enhanced functions not satisfied by existing wastewater treatment systems. For example, there is a long unmet need in the art for a simplified wastewater treatment process and apparatus that provides enhanced biological nutrient removal (BNR) and which, in certain embodiments, can produce class A bio-solids required for unrestricted land applications. In addition, there remains an unfulfilled need for wastewater treatment systems and methods that satisfy these expanded functions while minimizing the costs and environmental impacts that attend conventional wastewater treatment plant installation and operation.

Summary of Invention Paragraph (35):

[0034] The present invention provides devices and methods capable of treating residential waste water from a human population of 5,000--in a building about the size of a medium sized bungalow. This result may be achieved by devices and methods that produce recycle quality water, class A biosolids, and a clean odorless off gas.

Summary of Invention Paragraph (39):

[0038] In yet additional embodiments of the invention, a vertical shaft bioreactor is constructed and employed in novel designs and methods to achieve thermophilic aerobic digestion and pasteurization of sewage sludges, optionally including production of class A biosolids.

Brief Description of Drawings Paragraph (13):

[0051] FIG. 12 provides a graphical representation of the EPA time and temperature requirements for class A bio-solids.

Brief Description of Drawings Paragraph (14):

[0052] FIG. 13 provides an exemplary block flow diagram of the present invention adapted to produce recycle quality water, Class A bio-solids, and clean odourless off-gas. The following key applies to the FIG. 13:

Brief Description of Drawings - Table CWU (1):

1 Preliminary treatment A Fine screens B Solids hopper-Screenings and washed grit C Hyrdaclone degritter Waste water BNR treatment as described herein D Deoxygenation

unit [channel 32 + 40] E Denitrification [head tank 16] F Anoxic/anaerobic unit [channel 12] G Aerobic unit [zone 1 channel 80] H Nitrification [zone 2 head tank, 110 and 82] I Sedimentation clarifier [120] J Waste activated sludge float thickener K Alum or ferric chloride feeder L Process air compressor Recycle quality water [units required by law] M Flocculating tank N Cloth disk filter O Chlorination P Ultraviolet disinfection Q Backwash pump Thermophilic aerobic digestion as described herein class A biosolids R Zone 1 thermophilic aerobic digester S Zone 2 T Acid feeder U Polymer feeder V Centrifuge de-watering W Flotation cell X Air compressor Y Off gas collection system Z Class A bio-solids collection

Detail Description Paragraph (11):

[0062] e) Thermophilic aerobic digestion and pasteurization of sewage sludges to produce class A biosolids.

Detail Description Paragraph (40):

[0091] Within additional aspects of the invention, a novel nitrification process is provided which relies substantially or entirely upon residual dissolved oxygen originating near the bottom of zone 1 as the source of oxygen to drive the process. Yet another important benefit and distinction that arises by using the unspent gases from zone 1 in this fashion is the high level of CO.sub.2 available, which is also required by nitrifying bacteria as a source of inorganic carbon. In other nitrification systems, the primary inorganic carbon source depends on alkalinity of the wastewater and is typically determined by the presence of CaCO.sub.3. The bioreactor process systems of the invention are therefore more compact and require less energy than current, extended aeration systems. Bioreactors constructed and operated according to the invention also produce a better quality biomass (including class A biosolids if desired) that is easier to separate from the mother liquor.

Detail Description Paragraph (61):

[0112] Within yet another aspect of the invention, novel features and methods are provided for addressing the challenges involved in the disposal of by-product sludge and/or surplus bio-solids from the bioreactor 10 treatment processes. Recognizing the nutrient value of these biosolids, the EPA in the U.S. adopted 40 CFR 503 in 1993, which proscribes various process criteria to achieve class A bio-solids for unrestricted use as a soil supplement. Whenever possible, beneficial reuse of bio-solids is encouraged. One set of criteria for Class A bio-solids requires a minimum volatile solids reduction, as well as a Time-Temperature relationship, for example a 38% volatile Solids reduction and a 60.degree. C. temperature for 5 hours qualifies as a Class A product. FIG. 12 Within a modified embodiment of the invention, referring to FIG. 7, the bioreactor 10 is designed to function alternatively as a waste sludge digester and to meet the minimum volatile solids reduction and Time-Temperature relationship criteria for Class A biosolids production. In this regard, the reactor is specially designed and operated with a unique flow and zonal separation regime that provides for production of Class A biosolids in as little as 5-6 days, often in 3-4 days or less, using thermophilic bacteria operating at 58-65.degree. C. but typically 58.degree.-62.degree. C. and often 60.degree. C. The 38% volatile solids reduction is a measure of stability of the biomass or vector attraction reduction (VAR), while the elevated temperatures pasteurize the product to control E-coli and virtually eliminate salmonellae. Consuming 38% of the volatile matter minimizes odor potential and provides enough food energy for Thermophilic bacteria to raise the temperature of the reactor to over 60.degree. C., without applying exogenous heat.

Detail Description Paragraph (62):

[0113] Published data demonstrate two areas of concern for existing vertical shaft bioreactors that seek to produce class A biosolids (see, e.g., Report on VerTad operations King County WA, project 30900 May 20001, incorporated herein by reference.) First, small vertical bioreactors (e.g., "VerTad reactors", as described for example in U.S. patent application Ser. No. 09/570,162, filed May 11, 2000 (incorporated herein by reference), feature a relative disposition of zone 2 (polishing zone) below zone 1. These reactors have a comparatively large surface area to volume ratio, and excessive heat is lost to the surrounding geology. Small reactors therefore require supplemental heat to support class A biosolids production, which is available at additional cost by recapturing the waste heat from the compressor or from the hot effluent stream.

Detail Description Paragraph (63):

[0114] A second area of concern for previous vertical bioreactors directed to high quality biosolids production is that there is insufficient liquid to liquid separation between zones 1 and 2. Published data of tracer studies in VerTad reactors show that the zone 2 (polishing zone) behaves as a plug flow reactor, with a critical feature of localized back-mixing. Over a period of about 8 hours, zone 2 begins to mix with zone 1 and the whole system (zone 1 and zone 2) is mixed in 16-20 hrs. Accordingly, some solid particles, potentially containing salmonellae or other prohibited contaminants, can settle from zone 1 into zone 2 without being exposed to the required retention time at pasteurizing temperature to meet class A biosolids requirements.

Detail Description Paragraph (74):

[0125] Once sludge enters the reactor/digester 10' it has a mean residence time of approximately 2 to 3 days in zone 1, and 2 to 3 days in zone 2. The EPA criteria for the production of class A bio-solids dictates the time between batches, which varies with temperature--as an example the minimum residence time for a batch at 60.degree. C. is 5 hours, or about 4.8 batches per day. Therefore, zone 1 and zone 2 theoretically contain between 9.6 and 14.4 batches each. In practice, however, each batch would be about 8 hours, and therefore zone 1 and zone 2 would contain between 6 to 9 batches each. The overall residence time is determined by the biodegradability of the sludge. For class A bio-solids, the process must achieve a minimum of 38% volatile solids reduction which typically takes 3.5-5 days. The batching time is determined by the temperature (see, e.g., FIG. 12). The preferred operating temperatures of 58.degree. C.-62.degree. C. require approximately 8-4 hours.

Detail Description Paragraph (75):

[0126] As noted above, the air line 62 can be operated to maintain the air pressure in the air lock 172 of the reactor/digester 10' to control batching. Stopping the air flow in line 62 will also trigger a batch discharge after the appropriate processing time has elapsed. A batch can also be triggered by lowering the liquid level in the zone 2 head tank 15'. Once the batch in zone 2 is discharged, the head tank level in zone 1 is automatically lowered an equal amount by the action of the automatic batching valve located between the bottoms of zone 1 and 2, and the cycle repeats. When a batch is processed through the reactor, it is reduced in solids content from approximately 4-5% down to about 2-3%. This product (class A biosolids) may then be de-watered.

Detail Description Paragraph (76):

[0127] Published research by The University of Washington (Guild et al., Proceedings of WEF Conference, Atlanta Ga., 2001, incorporated herein by reference) indicates that when thermophic aerobic digested sludge from a vertical shaft reactor having certain features in common with the reactor of the present invention was fed to a mesophilic anaerobic digester, the retention time in the anaerobic digester was reduced, the overall volatile solids reduction was better, the dewaterability was better and required less polymer. The thermophilic aerobic digester is operated with a about a 2 day retention time and can generate enough heat to comply with Class A biosolids.

Detail Description Paragraph (78):

[0129] The selection of operating temperatures is very important in long, vertical thermophilic aerobic digesters because ammonium bicarbonate decomposes at about 60.degree. C. Ammonium bicarbonate is very important in the efficiency of the solids liquid separation (dewatering) step of the process. For instance, when operating a deep vertical thermophilic aerobic digester at 55.degree. C. to 58 .degree. C., the digested sludge samples were very granular before drying the sample but not after drying at about 104.degree. C. On one occasion when the head tank was opened without cooling the reactor (for emergency repair of a float switch), the inside surface, particularly the uninsulated access cover, was coated with tiny white angular crystals much like white sugar or salt. These crystals subsequently disappeared and were not found again at the higher operating temperatures. Another observation that is common, is that when a batch of product is transferred into the soak zone at about 58.degree. C. (where there is negligible biological activity), the temperature

increases and holds constant for about 2 hours, then cools at the cool-down rate of the reactor when operating on hot water. The heat of crystallization of 10,000 mg/L of ammonium bicarbonate would account for the apparent heat generated in the soak zone. Empirically, these observations would suggest the formation of ammonium bicarbonate crystals below 60.degree. C. This is contradicted by the fact that ammonium bicarbonate is very soluble in water, but less so in the presence of high levels of other dissolved solids, and perhaps the surface chemistry of the microbiology facilitate the crystallization process. For instance, Struvite (magnesium ammonium phosphate) is readily formed in anerobic digesters of plants using biological phosphorus removal but not in plants using chemical phosphorus removal. Controlling the reactor temperature to below 60.degree. C. may allow ammonium bicarbonate crystals to form which would easily float separate with the sludge.

Generate Collection

Print

Search Results - Record(s) 1 through 5 of 5 returned.

☐ 1. Document ID: US 20030121302 A1

L10: Entry 1 of 5

File: PGPB

Jul 3, 2003

PGPUB-DOCUMENT-NUMBER: 20030121302

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030121302 A1

TITLE: Production of a fertilizer product

PUBLICATION-DATE: July 3, 2003

INVENTOR - INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Oliver, Michael John Basil Pinetown ZA
Carsky, Milan Pinetown ZA
Hart, Oliver O?apos; Connor Pretoria ZA

US-CL-CURRENT: 71/11

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

KOMC

☐ 2. Document ID: US 20020148778 A1

L10: Entry 2 of 5

File: PGPB

Oct 17, 2002

KWIC

PGPUB-DOCUMENT-NUMBER: 20020148778

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020148778 A1

TITLE: Process and apparatus for conversion of biodegradable organic materials into

product gas

PUBLICATION-DATE: October 17, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Raven, Larry J. Fresno CA US

US-CL-CURRENT: 210/603

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Description

☐ 3. Document ID: US 6454944 B1

L10: Entry 3 of 5

File: USPT

Sep 24, 2002

US-PAT-NO: 6454944

DOCUMENT-IDENTIFIER: US 6454944 B1

TITLE: Process and apparatus for conversion of biodegradable organic materials into

product gas

DATE-ISSUED: September 24, 2002

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Raven; Larry J. Fresno CA 93703

US-CL-CURRENT: 210/603; 210/180



4. Document ID: US 5855664 A

L10: Entry 4 of 5

File: USPT

Jan 5, 1999

US-PAT-NO: 5855664

DOCUMENT-IDENTIFIER: US 5855664 A

TITLE: Solid waste landfill cover material and method of extending the useful life

of a solid waste disposal landfill

DATE-ISSUED: January 5, 1999

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Bielecki; Adam J. Avon Park FL Harrington; Robert Sebring FL

US-CL-CURRENT: $\frac{405}{129.9}$; $\frac{106}{18.11}$, $\frac{106}{18.13}$, $\frac{241}{DIG.38}$, $\frac{404}{82}$, $\frac{405}{129.25}$, $\frac{405}{129.95}$, $\frac{405}{129.95}$, $\frac{405}{129.19}$, $\frac{405}{129.25}$, $\frac{$



5. Document ID: US 5429750 A

L10: Entry 5 of 5

File: USPT

Jul 4, 1995

US-PAT-NO: 5429750

DOCUMENT-IDENTIFIER: US 5429750 A

TITLE: Method of treating wastewater sludges for pathogen removal and vector control

DATE-ISSUED: July 4, 1995

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Steele; Richard

Arden

NC

US-CL-CURRENT: 405/129.25; 210/764, 210/768, 422/33

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWAC
Drawi D	esc li	mage								
XXXIII XXIII X										
					Generate C	ollecti	ion	Print		

			Teri					Documer	14c	
	<u> </u>		reri	112				Documen	112	
	L7 no	ot 16								5
	$\ L\ $ no	ot 16								

Display Format: -

Change Format

Previous Page

Next Page

WEST						
	Generate Collection	Print				

L10: Entry 1 of 5

File: PGPB

Jul 3, 2003

DOCUMENT-IDENTIFIER: US 20030121302 A1 TITLE: Production of a fertilizer product

Summary of Invention Paragraph (4):
[0004] The EPA 40 CFR Part 503 standards include in particular criteria for biosolid disposal and category options for land application. Biosolids that meet the "Class A" pathogen reduction requirements in .sctn.503.32 (a) and the vector attraction reduction requirements in .sctn.503.33 (b) (1) - (b) (8), because of their low pollutant concentration and treatment to reduce pathogens to below detectable levels and reduced vector attraction, can be sold to the public without the restrictions required for land application of bio-solids. The U.S. EPA also promulgated rules to treat waste water sludge containing odour, animal viruses, pathogenic bacteria, and parasites, which will permit use of the waste water sludge as a fertilizer for agricultural lands and application directly to land as a dry granular material.

Generate Collection Print

L10: Entry 2 of 5

File: PGPB

Oct 17, 2002

DOCUMENT-IDENTIFIER: US 20020148778 A1

TITLE: Process and apparatus for conversion of biodegradable organic materials into product gas

Summary of Invention Paragraph (11):

[0011] An Upflow Anaerobic Sludge Blanket (UASB) process has been developed for bioconversion of feedstocks which contain primarily soluble organic waste wherein small amounts of solids, ordinarily less than 1 percent of the feedstock. The bacterial mass are allowed to settle in the reactor. The Upflow Anaerobic Sludge Blanket process and reactor are described in the following publications: G. Lettinga, et al, "Anaerobic Treatment of Methanolic Wastes," Water Research, Vol. 33, pp. 725-737, Pergamon Press Ltd. 1979; and G. Lettinga, et al, "Upflow Sludge Blanket Processes," 3rd International Symposium on Anaerobic Digestion, 1983, Cambridge Mass.; and G. Lettinga, et al, "Anaerobic Treatment of Raw Domestic Sewage" at Ambient Temperatures Using a Granular Bed .UASB Reactor, Biotechnology and Bioengineering, Vol. XXV, pp. 1701-1723, 1983. This reactor design is limited to liquid feedstocks containing less than about 1 percent solids, and it requires effective gas/liquid separators, recycle for bed expansion, and means for distributing the feed over the bottom of the reactor.

Summary of Invention Paragraph (33):

[0032] In another aspect of the invention, an appropriately selected feedstock concentration is mixed with a waste stream and then introduced into the digester(s) for conversion into biogas. Combining the system with a municipal or other waste water treatment facility will help to create a high volume of biogas which is profitable to sell, while also helping the facility to attain the <u>Class A biosolids</u> 40 C.F.R. Part 503 Standard by pasteurizing sewage sludge.

Summary of Invention Paragraph (38):

[0037] It is an object of this invention to aid municipal wastewater treatment plants in meeting the fecal coliform requirements of the recent 40 C.F.R. Part 503 Standards for Class A biosolids through the pasteurization capability of the thermophilic digester.

Generate Collection Print

L10: Entry 3 of 5 File: USPT Sep 24, 2002

DOCUMENT-IDENTIFIER: US 6454944 B1

TITLE: Process and apparatus for conversion of biodegradable organic materials into

product gas

Brief Summary Text (11):

An Upflow Anaerobic Sludge Blanket (UASB) process has been developed for bioconversion of feedstocks which contain primarily soluble organic waste wherein small amounts of solids, ordinarily less than 1 percent of the feedstock. The bacterial mass are allowed to settle in the reactor. The Upflow Anaerobic Sludge Blanket process and reactor are described in the following publications: G. Lettinga, et al, "Anaerobic Treatment of Methanolic Wastes," Water Research, Vol. 33, pp. 725-737, Pergamon Press Ltd. 1979; and G. Lettinga, et al, "Upflow Sludge Blanket Processes," 3rd International Symposium on Anaerobic Digestion, 1983, Cambridge Mass.; and G. Lettinga, et al, "Anaerobic Treatment of Raw Domestic Sewage" at Ambient Temperatures Using a Granular Bed UASB Reactor, Biotechnology and Bioengineering, Vol. XXV, pp. 1701-1723, 1983. This reactor design is limited to liquid feedstocks containing less than about 1 percent solids, and it requires effective gas/liquid separators, recycle for bed expansion, and means for distributing the feed over the bottom of the reactor.

Brief Summary Text (33):

In another aspect of the invention, an appropriately selected feedstock concentration is mixed with a waste stream and then introduced into the digester(s) for conversion into biogas. Combining the system with a municipal or other waste water treatment facility will help to create a high volume of biogas which is profitable to sell, while also helping the facility to attain the Class A biosolids 40 C.F.R. Part 503 Standard by pasteurizing sewage sludge.

Brief Summary Text (38):

It is an object of this invention to aid municipal wastewater treatment plants in meeting the fecal coliform requirements of the recent 40 C.F.R. Part 503 Standards for <u>Class A biosolids</u> through the pasteurization capability of the thermophilic digester.

Other Reference Publication (1):

G. Lettinga et al, Anaerobic Treatment of Raw Domestic Sewage at Ambient Temperature Using a <u>GranularBed</u> UASB Reactor, Biotechnology and Bioengineering, vol. XXV p. 1701-1723 (1983).

Generate Collection Print

L10: Entry 4 of 5 File: USPT Jan 5, 1999

DOCUMENT-IDENTIFIER: US 5855664 A

TITLE: Solid waste landfill cover material and method of extending the useful life

of a solid waste disposal landfill

Detailed Description Text (98):

Depending upon field conditions such as rain, snow, etc., a <u>granular</u> dry fertilizer mixture may be substituted for the water soluble nutrient/fertilizer. The <u>granular</u> dry fertilizer mixture can be applied by using a rotary fertilizer spreader or its equivalent. Biosolid fertilizer (domestic sewage sludge) material as regulated by the Federal Environmental Protection Agency (FEPA) under Code of Federal Regulations (CFR) Part 503, Standards for the Use or Disposal of Sewage Sludge and/or its equivalent may also be used.

Detailed Description Text (99):

Class A (Grade I) domestic waste water residuals (sludge), Class B (Grade II) domestic waste water residuals (sludge) when utilized as biosolid fertilizer material and regulated by the State of Florida's Administrative Code (F.A.C.) Chapters 62-640 and/or its equivalent can be used also. Additionally, a controlled time release fertilizer can be used. The actual fertilizer materials to attain optimal levels of the primary nutrients Nitrogen, Phosphorous and Potassium (NPK) can be but are not limited to, nitrogen (amines, nitrates, ammonia salts and the like), phosphorous-phosphates (calcium, super phosphate, ammonium phosphate and the like), potassium (potassium sulfate, potassium nitrate, potassium carbonate and the like). Additionally, secondary nutrients (trace elements) such as boron, copper, iron, manganese, molybdem, zinc and the like should be also added. Adjustments to the levels of primary nutrients, secondary nutrients (trace metals) as well as the pH levels are made as often as required to maintain optimal nutrient conditions for rapid biodegradation/decomposition of the applied C&D-RSM daily/initial product layer. Caution should be exercised in applying the nutrient mixtures to the disposed municipal solid waste (MSW) material. Concentrated nutrient application may accelerate excessive biodegradation, resulting in spontaneous combustion of the "covered" MSW disposal material. Furthermore, depending upon the amount of biodegradable organic material contained within the solid waste disposal material received at the MSW disposal landfill, the addition of nutrients (fertilizer) may not be required. All of these elements combine to create a manufactured product with known specification which satisfy known requirements. Additionally, this product is reproduced in such a way that a guarantee can be issued.

End of Result Set

Generate Collection Print

L10: Entry 5 of 5

File: USPT

Jul 4, 1995

DOCUMENT-IDENTIFIER: US 5429750 A

TITLE: Method of treating wastewater sludges for pathogen removal and vector control

Brief Summary Text (3):

This invention relates generally to a process for the treatment of wastewater sludges and biosolids in order to achieve the highest classification (Class A) for sludge and sludge products with respect to pathogen removal and vector control and in particular, to an improved process for wastewater treatment that utilizes ambient and internal sludge pressure variation such as vacuum and high pressure extrusion in conjunction with pH conditioning to achieve U.S.E.P.A. Class A sludge quality in conjunction with reducing the effects of putrescible organic compounds in achieving vector attraction reduction.

Brief Summary Text (13):

A method for treating wastewater sludge and biosolids to achieve Class A pathogen reduction standards and to reduce vector attraction, comprising the steps of providing a predetermined amount of dewatered sludge and as a first step, raising the sludge pH to above 12 by adding alkaline reagents such lime, cement, cement kiln dust (ckd), fly ashes, or similar caustic agents.

Detailed Description Text (2):

Referring now to the drawings and in particular, FIG. 1, a method is disclosed of treating sludge or biosolids which are wastewater sludge in order to achieve Class A PFRP pathogen removal and at the same time reducing vector attraction.

Detailed Description Text (16):

One of the methods that can be used to meet pathogen reduction and vector control for Class A sludge is drying, which is greatly improved when finely ground paper fluff is homogeneously mixed with the sludge. The fibers form an overlapping network extending throughout this material. Paper fibers within the mass wick the moisture from the center, especially if the sludge is formed into relatively uniform pellets or granules.

Generate Collection

Print

Search Results - Record(s) 1 through 8 of 8 returned.

☐ 1. Document ID: US 20020162795 A1

L11: Entry 1 of 8

File: PGPB

Nov 7, 2002

PGPUB-DOCUMENT-NUMBER: 20020162795

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020162795 A1

TITLE: Methods and apparatus for biological treatment of waste waters

PUBLICATION-DATE: November 7, 2002

INVENTOR-INFORMATION:

NAME

CITY

STATE

COUNTRY

RULE-47

Pollock, David C.

Calgary

CA

US-CL-CURRENT: 210/621; 210/626

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

KMC

☐ 2. Document ID: US 20020098982 A1

L11: Entry 2 of 8

File: PGPB

Jul 25, 2002

PGPUB-DOCUMENT-NUMBER: 20020098982

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020098982 A1

TITLE: Production and use of biosolid granules

PUBLICATION-DATE: July 25, 2002

INVENTOR-INFORMATION:

NAME

CITY

STATE

RULE-47

Burnham, Jeffrey C.

Naples

FL

US

COUNTRY

US-CL-CURRENT: 504/359; 504/117, 504/367, 71/64.02, 71/64.11

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draws Description

KWC

☐ 3. Document ID: US 6521133 B1

L11: Entry 3 of 8

File: USPT

Feb 18, 2003

US-PAT-NO: 6521133

DOCUMENT-IDENTIFIER: US 6521133 B1

TITLE: Process for thermal sludge disinfection

DATE-ISSUED: February 18, 2003

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Roediger; Markus Sewickley PA

US-CL-CURRENT: 210/742; 210/175, 210/744, 210/764, 210/774, 210/916, 422/1, 422/38

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

4. Document ID: US 6399359 B1

L11: Entry 4 of 8 File: USPT Jun 4, 2002

US-PAT-NO: 6399359

DOCUMENT-IDENTIFIER: US 6399359 B1

TITLE: Composition system

DATE-ISSUED: June 4, 2002

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Hofstede; Harrie West Leederville. W.A. 6007

US-CL-CURRENT: 435/262.5; 435/290.1, 435/290.4, 71/9

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC |
Draw, Desc | Image |

5. Document ID: US 6256902 B1

L11: Entry 5 of 8 File: USPT Jul 10, 2001

US-PAT-NO: 6256902

DOCUMENT-IDENTIFIER: US 6256902 B1

TITLE: Apparatus and method for desiccating and deagglomerating wet, particulate

materials

DATE-ISSUED: July 10, 2001

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Flaherty; John R. Philadelphia PA 19106 Gehring; Bradley P. Hudson WI 54016

US-CL-CURRENT: 34/379; 110/222, 110/224, 210/609, 210/769, 210/771, 34/381, 34/384,

34/387, 34/61, 34/68

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC |
Draw Desc Image

☐ 6. Document ID: US 6206091 B1

L11: Entry 6 of 8

File: USPT

Mar 27, 2001

US-PAT-NO: 6206091

DOCUMENT-IDENTIFIER: US 6206091 B1

TITLE: Process and apparatus for treating waste

DATE-ISSUED: March 27, 2001

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Buehler; Verne T.

Waukesha

kesha WI

US-CL-CURRENT: 165/143; 165/108, 210/609

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

KOMC

7. Document ID: US 6047768 A

L11: Entry 7 of 8

File: USPT

Apr 11, 2000

US-PAT-NO: 6047768

DOCUMENT-IDENTIFIER: US 6047768 A

TITLE: Process and apparatus for treating waste

DATE-ISSUED: April 11, 2000

INVENTOR-INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

Buehler, III; Verne T.

Waukesha

WI

US-CL-CURRENT: 165/143; 165/108, 210/609

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc. Image.

KONAC

1 8. Document ID: US 5855664 A

L11: Entry 8 of 8

File: USPT

Jan 5, 1999

US-PAT-NO: 5855664

DOCUMENT-IDENTIFIER: US 5855664 A

TITLE: Solid waste landfill cover material and method of extending the useful life

of a solid waste disposal landfill

DATE-ISSUED: January 5, 1999

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Bielecki; Adam J.

Avon Park

FL

Harrington; Robert

Sebring

FL

Full Title	e Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMC
Draw. Desc	Image								
				Generate C	Collecti	ion	Print		
			accommon my		encontrolli (in anti dibitati de c	manufacture de la constitución d			
		Town					Daguman	.45	
<u> </u>		Term	15				Documen	112	
14 a	ınd 12								8
				- 11					11

Display Format: - Change Format

Previous Page Next Page

Jul 25, 2002

WEST

Generate Collection Print

File: PGPB

PGPUB-DOCUMENT-NUMBER: 20020098982

PGPUB-FILING-TYPE: new

L11: Entry 2 of 8

DOCUMENT-IDENTIFIER: US 20020098982 A1

TITLE: Production and use of biosolid granules

PUBLICATION-DATE: July 25, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Burnham, Jeffrey C. Naples FL US

APPL-NO: 10/ 051387 [PALM]
DATE FILED: January 22, 2002

RELATED-US-APPL-DATA:

Application is a non-provisional-of-provisional application 60/262631, filed January 22, 2001,

Application is a non-provisional-of-provisional application 60/272469, filed March 2, 2001,

INT-CL: [07] <u>A01</u> <u>N</u> <u>25/28</u>, <u>A01</u> <u>N</u> <u>25/00</u>, <u>A01</u> <u>N</u> <u>63/00</u>, <u>A01</u> <u>N</u> <u>25/12</u>

US-CL-PUBLISHED: 504/359; 504/117, 504/367, 71/64.02, 71/64.11 US-CL-CURRENT: 504/359; 504/117, 504/367, 71/64.02, 71/64.11

ABSTRACT:

This invention relates to the production and use of encapsulated and/or concentrically-constructed fertilizer or bioremediation granules such as, for example, granules of 0.5 mm to 10 mm in diameter constructed so that there are at least two components to the granule including a core with a surrounding capsule or a core with one or more concentric <u>layers</u> that are distinguishable from the core with respect to nutrient content, density, hardness, solubility, composition, microbial content and permeability, as in permeability to odors or the permeability of nutrients that might volatize to the atmosphere or leach into the soil. The basic idea was to create a method for manufacturing and using fertilizer granules, which incorporate multiple concentric layers or a core plus an encapsulating outer layer.

Generate Collection Print

L11: Entry 3 of 8 File: USPT Feb 18, 2003

DOCUMENT-IDENTIFIER: US 6521133 B1

TITLE: Process for thermal sludge disinfection

Brief Summary Text (2):

The present invention is related to thermal disinfection or pasteurization of liquid sludge, such as sewage sludge. Disinfection is the inactivation of pathogens to levels low enough so that the sludge can be beneficially used, e.g. for land application, without infection risks. Pasteurization is the disinfection of liquids by heat. Thermal disinfection requires maintenance of every sludge particle at a certain temperature T (above 50.degree. C.) for a minimum time period t. The time period t depends on the temperature T, but should not be less than 30 minutes for sewage sludge, even if T is 70.degree. C. or above. The Environmental Protection Agency (EPA) of the United States of America has published time-temperature relationships for thermal sludge treatment in Biosolids Rule 503. The present invention proposes a continuous flow system for thermal sludge disinfection complying with the EPA regulations and providing so-called Class A Biosolids.

Brief Summary Text (14):

The flow pattern within the chambers can also be influenced by thermal convection. If the incoming sludge has a slightly higher temperature than the sludge in the chamber, the incoming sludge stratifies at the top and moves down in a <u>layer</u> as it cools down. Because the flow has approximately a plug-flow characteristic in this case, the minimum detention time is only slightly shorter than the mean detention time. It is therefore an advantage to have a downward flow.

Detailed Description Text (17):

With a sludge temperature in chambers 10 and 14 of 70.degree. C., the minimum sludge detention time in the chambers is 30 minutes, according to EPA requirements, to guarantee sufficient pathogen reduction to achieve Class A biosolids. Considering a safety factor of 2, the volume of the chambers must be minimum 2 m3 to provide an average detention time of 1 hour in the chambers. Tank 99 including the chambers 10 and 14 could have a diameter of 0.8 m and a cylindrical height of 5 m. This height also includes a freeboard of around 1 m. The hydraulic diameter for vertical flow through chambers 10 and 14 is around 0.5 m. The ratio of the flow path length through both chambers to the hydraulic diameter of the chambers is about 16:1. This is a high ratio and sufficient to prevent short circuits.

Generate Collection | Print |

L11: Entry 4 of 8 File: USPT Jun 4, 2002

DOCUMENT-IDENTIFIER: US 6399359 B1

TITLE: Composition system

Brief Summary Text (34):

Product compost which may typically have less than ten (10) total coliforms per gram compost in comparison with ARMCANZ guidelines (see Agriculture and Resource Management Council of Australia and New Zealand Water Technology Committee, Guidelines for Sewage Systems—Biosolids Management Occasional Paper WTC No 1/95 October, 1995) which specify that Class A compost may contain a maximum pathogen concentration of 100 thermotolerant coliforms per gram compost.

Detailed Description Text (24):

The ARMCANZ guidelines (see Agriculture and Resource Management Council of Australia and New Zealand Water Technology Committee, Guidelines for Sewage Systems--Biosolids Management Occasional Paper WTC No 1/95 October, 1995) require composting to be carried out at thermophilic temperatures (>55.degree.C.) for at least three continuous days to produce grade 1A compost. The ARMCANZ grade 1A composting requirements were met throughout the windrow using the method and system of the invention, including at the windrow surface. Reference is made to a FIG. 3 which shows a temperature profile at the windrow surface and various depths within the pile. It has been found in previous practice that in open windrow composting systems the surface temperatures are too low (near ambient, at 15-30.degree. C.) to meet these guidelines. It is thought, without wishing to be bound by any theory, that the preferably flexible cover of the system insulates the windrow from climatic conditions thereby preventing heat loss. Materials for the cover may be selected with this in view. Such a cover may also trap solar radiation which may assist in heating the windrow surface. It has been found that windrow surface temperatures may reach up to 70.degree. C. during composting and this a special advantage of the present system. An air layer containing trapped air will typically be present and acids in insulation of the compost mass.

CLAIMS:

3. The method of claim 1 wherein the environment is substantially filled with compostable material leaving an insulating \underline{layer} of air between the material and the cover.

Generate Collection Print

L11: Entry 5 of 8

File: USPT

Jul 10, 2001

DOCUMENT-IDENTIFIER: US 6256902 B1

TITLE: Apparatus and method for desiccating and deagglomerating wet, particulate

materials

Brief Summary Text (10):

"Land application for beneficial use" is the application of biosolids to land, either to condition the soil, or to fertilize crops or other vegetation grown in the soil. Biosolids can be beneficially land-applied on agricultural land, forest land, reclamation sites, golf courses, public parks, roadsides, plant nurseries and home land and gardens. Under the 503 Regulations, biosolid products that meet stringent requirements, including sufficiently low concentrations of certain pathogens and pollutants, and minimal attractiveness to disease vectors such as insects and rodents, are considered by the EPA to be Class A, "Exceptional Quality" biosolids. Class A biosolids are treated by the EPA in the same manner as common fertilizers; thus, they are exempt from federal restrictions on their agricultural use or land application. Biosolids falling short of the highest EPA standards may nevertheless qualify as Class B biosolids.

Brief Summary Text (37):

Ultimately, the inventive apparatus and method are capable of converting common municipal sludge into a 90% to 98%-dry, Class A biosolid, beneficially reusable as a fertilizer, filler or fuel. And, this is accomplished at roughly half the capital cost of competitive direct and indirect-drying systems. Also, as there is a significant associated weight reduction between the raw and final products, the apparatus and method disclosed and claimed herein significantly lower biosolid shipping, handling, and landfill costs.

Brief Summary Text (38):

All of the forgoing attributes combine in the inventive sludge drying apparatus and method such that, when rendered in the form of a plant able to process 10 tons of 25%-dry sludge per hour, the yield is approximately 63 tons of 90%-dry Class A biosolid per day, at a cost of approximately \$118 per dry ton.

Detailed Description Text (43):

Ultimately, in either embodiment, the material remains in the circuit until the maximum possible amount of surface moisture has been exposed to dry air, and removed. Particles of a size approximately 60 microns, and under, are drawn out of toroidal ring dryer 38 with the process air as it flows into product output duct 62. Output duct 62's upstream end is mated with toroidal ring dryer 38's innermost wall 110, thus causing duct 62 to draw off the lightest, finest particles circulating in toroidal ring dryer 38. Exhaust fan 70 draws the majority of the process air from product output duct 62 upward through baghouse 64. The fine, dry particles 106 suspended in process air flow to baghouse 64, where a majority drop out into collection bin 78. The finest particles get drawn upward into baghouse 64's Nomex bags (not shown). As suspended particles build up into layers of any substantial thickness on the inner surfaces of the Nomex bags, they tend to reduce air flow through the bags' fabric, thus permitting the buildup to drop out into collection bin 78. Particles as small as 2.5 microns are filtered out of the process air by baghouse 64.

Detailed Description Text (44):

A portion of the dried sludge from collection bin 78 is carried away by product

take-away conveyor 80. Product take-away conveyor 80 may terminate at, and deliver its output to, for example, receiving truck 114. The dried sludge product trucked away is a 90% to 98%-dry, Class A biosolid, usable as a fertilizer, filler or fuel.

Detailed Description Text (88):

In a fifth embodiment of the invention, shown in its diagrammatic essence in FIG.7 and identified with reference numeral 500, yet another arrangement employs an alternative to a ring dryer. This embodiment also uses the pneumatic friction drying concept, passing sludge with high-speed air through an elongate shredding conduit 502 to effect preliminary shredding and desiccation. One or more sweeping 90-degree bends 504 in this elongate shredding conduit are also preferred. The partially-processed product issuing from the terminal, downstream end 506 of the elongate shredding conduit 502 is broadcast onto a wire mesh belt conveyor 508, thus coating the wire mesh belt. Mesh belt 508 runs in a low-pressure drying chamber 510, with flowing, heated process air from blowers 512 beneath mesh belt 508 blowing through the thin coat of material on the belt. The length of drying chamber 510 is preferably sufficient to permit the material at the end of belt 508 and drying chamber 510 to be 90% dry.

Generate Collection Print

L11: Entry 6 of 8 File: USPT Mar 27, 2001

DOCUMENT-IDENTIFIER: US 6206091 B1

TITLE: Process and apparatus for treating waste

Brief Summary Text (7):

Some heat recovery systems may employ a stainless steel tank placed inside of a larger stainless steel tank wherein each tank acts as a solid waste batch storage tank while heat is being transferred through the wall between the two sludge tanks. The heat is being transferred between the hot solid waste and cold solid waste without the use of an intermediate water media. Often, mechanical mixers are used to agitate the solid waste in each tank to facilitate the heat transfer. These heat recovery systems have very high fabrication costs, are very complex, have high ceiling requirements, and are difficult to maintain and clean. Additionally, the mechanical mixers have the further drawback of becoming clogged over time. As the mixers become clogged or as sludge scale coats the walls of the chambers, it becomes necessary to disassemble and clean the tanks to restore adequate heat transfer efficiency. Disassembly shuts down the whole system.

Detailed Description Text (63):

The first stage of the apparatus 10 requires no biological activity. The thermal conditioning at temperatures and for periods of time that are determined empirically in the first stage results in pathogen reduction as well as solubilization of solids. Temperatures and times are selected so as to obtain pathogen reduction without biological digestion. Hence, it is appreciated that the first stage of the apparatus 10 is greatly simplified and requires less user attention. The user need not monitor the process of the first stage or generally make operating adjustments to apparatus 10 to maintain the viability of a microbiological population for solids degradation. Generally, it is preferred that the operating temperature of the first stage be maintained at about 150.degree. F. for about 1 hour. The time and temperature requirements generally are dictated by those requirements necessary for pathogen reduction to meet Class A biosolids in U.S. government 503 regulations.

Detailed Description Text (64):

Once pathogens have been reduced to meet Class A criteria, the preconditioned, solubilized solids are stabilized in anaerobic digester 112. Anaerobic digester 112 involves a two step process. Stabilization is generally defined as an overall reduction of volatile matter in the solids of about 38 percent. This level of stabilization meets the criteria for Class A biosolids as specified in Option 1 of U.S. government 503 regulations. As described above, methane gas generated in the second stage is used to maintain the operating temperature of stage one. Hence, an optional outside fuel source is generally not required. Therefore, with respect to operating temperature, the process is autothermal.

Detailed Description Text (92):

The overall process is thermal conditioning followed by anaerobic digestion in digester 112. The addition of tank for thermal conditioning 54 and heat exchanger 42 constitute the main components to retrofit an existing anaerobic digestion system into one capable of producing Class A Biosolids.

End of Result Set

Generate Collection Print

L11: Entry 8 of 8

File: USPT

Jan 5, 1999

DOCUMENT-IDENTIFIER: US 5855664 A

TITLE: Solid waste landfill cover material and method of extending the useful life of a solid waste disposal landfill

Detailed Description Text (26):

In present day practice, the disposed compacted and leveled MSW material is covered at the end of the business day by spreading a <u>layer</u> of dirt, sand, earthen material or the like over the exposed portion (working face) of the waste material. The dirt, sand or earthen material is utilized as daily/initial cover material. Additionally, native dirt or the like taken from on or near the MSW disposal landfills geographic site boundaries may also be used. Furthermore, because of the large quantities needed and the expense incurred in its excavation and transportation to the MSW disposal landfills working face, the dirt, sand, earthen material or the like are considered commercial products in this related art. The thickness of this cover layer is primarily dependent upon the length of time the leveled and compacted waste material in the MSW disposal landfill is to remain temporarily covered. This cover layer, however, must be at least six inches in thickness to satisfy environmental regulatory agency requirements.

Detailed Description Text (27):

In usual practice, the waste material placed in a Municipal Solid Waste (MSW) disposal landfill is leveled, compacted and then covered with daily/initial cover material for a short period of time, typically overnight. If it is necessary to cover the disposed MSW for a longer period of time, a thicker layer of daily/initial cover material must be applied. The primary problem associated with the use of dirt, sand or earthen material is that large quantities of the cover material are necessary to cover the disposed municipal solid waste. Because dirt, sand or earthen material do not decompose/biodegrade or compress to any great degree, there is a substantial loss of available volume for waste, which is forever occupied by the daily applied layers of dirt, sand, earthen material or the like. This is particularly a problem with MSW disposal landfill that are required to be covered at the end of each day of operation. In this instance, a layer of space six inches in depth across the entire landfill working face is lost everyday to the dirt, sand, earthen material or the like which will forever occupy this space. This loss of available space in the landfill, not to mention the cost of the cover material, results in substantial revenue losses to landfill operators. The manufactured Construction and Demolition (C&D) disposal debris RSM product serves as a substitute specification particle sized material replacing the normally used dirt, sand, earthen or the like daily cover material.

Detailed Description Text (29):

In accordance with the first embodiment, at the subtitle "D" MSW disposal landfill, Class I solid waste disposal landfill, MSW disposal landfill operating within a slurry wall leachate containment system or other approved site location, the received MSW is compacted and leveled into place at the end of the business day. Next, a <u>layer</u> of manufactured C&D disposal debris RSM product, which is the "actual" substitute/alternative daily cover material <u>layer</u> is mechanically applied/spread on top of the compacted and leveled disposed municipal solid waste until the required six inch thickness is achieved. The C&D-RSM product which is applied over the top portion of the disposed MSW substitutes/replaces the normally used dirt, sand or earthen daily/initial cover material. When applied to a typical 5,000 square foot

working face area of a MSW disposal landfill 125-150 tons of C&D-RSM product are applied. Equipment utilized for spreading the C&D disposal debris RSM product can be, for example, a dozer, wheel loader, self-loading and/or unloading pan or the like. The proper concentration of formulated solution is next applied by spray method soaking the applied RSM product layer until it becomes well saturated. It is at this point, when the C&D disposal debris RSM product is soaked with the formulated solution, that it becomes acceptable and approved daily/initial cover material.

<u>Detailed Description Text</u> (35):

The boric acid in the formulated solution serves two distinct purposes. First, the boric acid acts as a disinfectant temporarily controlling any potential disease producing micro-organisms which may be contained within the C&D-RSM substitute daily cover product Layer. Furthermore, any disease producing organisms which may be present on any de minimis amounts of municipal solid waste which may protrude from the applied daily cover material are also temporarily disinfected. Additionally, any disease producing micro-organisms which may be travelling upwards with vapors and gasses as they migrate through the disposed municipal solid waste and escape to the surface must travel through this treated C&D-RSM daily cover Layer and are also temporarily disinfected. Therefore, the potential disease producing micro-organisms are controlled at their source before they can be carried or spread by vectors to humans. This satisfies the environmental regulatory authority daily cover requirement of controlling disease vectors.

Detailed Description Text (36):

Even though adverse field conditions may be encountered such as rain, snow, ice or similar high moisture climate conditions, the formulated solution must still be applied to control potential disease producing micro-organisms and odors. Disease vectors are rodents, flies, mosquitoes, or other animals, including insects, capable of transmitting disease producing organisms to humans. The manufactured C&D disposal debris RSM product contains no source of putrescible material (food) or other items with which to attract rodents, flies, mosquitoes, animals or insects. The boric acid in the formulated solution also acts to temporarily deter insects away from this treated C&D-RSM substitute daily/initial cover layer.

Detailed Description Text (37):

Secondly, the boric acid in the formulated solution acts as a temporary fire retardant coating all of the three inch or less in size particles of C&D-RSM product which it encounters and remains attached to them after the solution dries. This, in turn, reduces the fire potential of any combustible solid particles contained within the C&D-RSM daily/initial cover product. The boric acid in the formulated solution enables the C&D-RSM product to satisfy the environmental regulatory authority requirement of controlling fires. Furthermore, after treatment with the formulated solution of the related art, the substitute RSM daily cover product has the ability to pass the accepted ASTM test method D 4982-89.

Detailed Description Text (56):

The present invention contemplates the use of other fire retardants for application to the C&D disposal debris RSM daily/initial cover product. Various fire retardants when applied to the C&D disposal debris RSM substitute daily cover product layer include the following:

Detailed Description Text (94):

The detergent in the formulated solution breaks the surface tension of the water allowing the chemical values of this prepared mixture to evenly <u>coat</u> all of the particles of the manufactured C&D RSM substitute daily cover product. The detergent should be non-foaming biodegradable, contain no bleach or fragrance and be compatible with boric acid and the present nutrients.

Detailed Description Text (95):

After serving its purpose of covering the disposed municipal solid waste for the required one day time period this completed daily/initial cover layer of treated C&D disposal debris RSM product is buried under garbage, putrescible household waste, rubbish or the like on the next day of landfill operation.

Detailed Description Text (96):

The decomposition/biodegradation of the buried C&D disposal debris RSM daily/initial cover product <u>layer</u> proceeds by utilizing the natural occurring microbes available from the disposed municipal solid waste which trickle down to this treated region.

Detailed Description Text (99):

Class A (Grade I) domestic waste water residuals (sludge), Class B (Grade II) domestic waste water residuals (sludge) when utilized as biosolid fertilizer material and regulated by the State of Florida's Administrative Code (F.A.C.) Chapters 62-640 and/or its equivalent can be used also. Additionally, a controlled time release fertilizer can be used. The actual fertilizer materials to attain optimal levels of the primary nutrients Nitrogen, Phosphorous and Potassium (NPK) can be but are not limited to, nitrogen (amines, nitrates, ammonia salts and the like), phosphorous-phosphates (calcium, super phosphate, ammonium phosphate and the like), potassium (potassium sulfate, potassium nitrate, potassium carbonate and the like). Additionally, secondary nutrients (trace elements) such as boron, copper, iron, manganese, molybdem, zinc and the like should be also added. Adjustments to the levels of primary nutrients, secondary nutrients (trace metals) as well as the pH levels are made as often as required to maintain optimal nutrient conditions for rapid biodegradation/decomposition of the applied C&D-RSM daily/initial product layer. Caution should be exercised in applying the nutrient mixtures to the disposed municipal solid waste (MSW) material. Concentrated nutrient application may accelerate excessive biodegradation, resulting in spontaneous combustion of the "covered" MSW disposal material. Furthermore, depending upon the amount of biodegradable organic material contained within the solid waste disposal material received at the MSW disposal landfill, the addition of nutrients (fertilizer) may not be required. All of these elements combine to create a manufactured product with known specification which satisfy known requirements. Additionally, this product is reproduced in such a way that a guarantee can be issued.

Detailed Description Text (132):

In a preferred process method of this second embodiment, the construction and demolition disposal debris raw material/feed stock is delivered to the manufacturing site. The Class III/commercial solid waste raw material/feed stock is delivered to the same manufacturing site. At the permitted/licensed or approved manufacturing/processing site, both of these raw material/feed stocks are fed simultaneously into a shredder equipped with three inch openings in its discharge grates. The resulting commingled inseparable mixture of shredder output materials is the manufactured C&D-Class III recovered screened materials substitute daily cover product of this related embodiment. After application of this substitute daily/cover product over disposed municipal solid waste, it is treated by spray method with the formulated solution until the desired conditions are achieved. This treatment enables this applied product to satisfy the specific environmental regulatory authority "Daily Cover Requirements" and qualify as an approved substitute daily/initial cover material. This completed substitute daily/initial cover product layer of combined Class III and C&D debris RSM products are buried under municipal solid waste material, i.e., garbage, putrescible household waste, refuse, rubbish or the like on the next day of landfill operation. As this completed cover material layer decomposes/biodegrades, valuable landfill disposal space is regained.

Detailed Description Text (144):

After application of this substitute daily/cover product over disposed municipal solid waste, it is treated by spray method with the formulated solution until the desired conditions are achieved. This treatment enables this applied product to satisfy the specific environmental regulatory authority "Daily Cover Requirements" and qualify as an approved substitute daily/initial cover material. This completed substitute daily/initial cover product layer of combined automobile shredder residue and C&D debris RSM products are buried under municipal solid waste material, i.e., garbage, putrescible household waste, refuse, rubbish or the like on the next day of landfill operation. As this completed cover material layer decomposes/biodegrades, valuable landfill disposal space is regained.

Detailed Description Text (173):

After application of this substitute daily/initial product over disposed municipal solid waste, it is treated by spray method with the formulated solution until the

desired conditions are achieved. This treatment enables this applied product to satisfy the specific environmental regulatory authority "Daily Cover Requirements" and qualify as an approved substitute daily/initial cover material. This completed substitute daily/initial cover product layer of combined municipal solid waste RSM and construction and demolition disposal debris RSM product are buried under municipal solid waste, i.e., garbage, putrescible household waste, refuse or the like on the next day of landfill operation. As this completed cover material layer decomposes/biodegrades, valuable landfill disposal space is regained thus extending the useful life of a solid waste disposal landfill utilizing this substitute daily/initial cover product.

CLAIMS:

1. A method of extending a useful life of a solid waste disposal landfill comprising the steps of:

providing a raw material feed stock composed of a commingled inseparable mixture of solid waste components;

recovering a screened material product from said raw material feed stock for use as a substitute daily/initial solid waste disposal landfill cover material;

periodically leveling and compacting disposed waste material in the solid waste disposal landfill;

applying a <u>layer</u> of said recovered screened material product to the leveled and compacted disposed waste material; and

spraying and soaking said <u>layer</u> of recovered screened material product with a solution to inhibit combustion and to promote decomposition consisting of:

water;

boric acid;

detergent;

- a nutrient; and
- a deodorant.
- 5. A method as recited in claim 3 wherein said step of spraying and soaking includes soaking said applied <u>layer</u> with said solution until said <u>layer</u> has a moisture content of at least 25%.
- 6. A method of extending a useful life of a solid waste disposal landfill comprising the steps of:

providing a raw material feed stock composed of a commingled mixture of solid waste components;

recovering a screened material product from said raw material feed stock for use as a substitute daily/initial solid waste disposal landfill cover material;

periodically leveling and compacting disposal waste material in the solid waste disposal landfill;

applying a $\underline{\text{layer}}$ of said recovered screened material product to the leveled and compacted waste material; and

spraying and soaking said \underline{layer} of recovered screened material product with a solution to inhibit combustion and to provide a nutrient to promote decomposition.

10. A method as recited in claim 8 wherein said step of spraying and soaking includes soaking said applied layer with said solution until said layer has a

moisture content of at least 25%.

- 11. A method as recited in claim 10 wherein said recovered screen material product is sprayed and soaked with said solution and stored prior to said step of applying said <u>layer</u> wherein said stored recovered screen material is available for subsequent use as said substitute daily/initial solid waste disposal landfill cover material.
- 13. A method of extending a useful life of a solid waste disposal landfill comprising the steps of:

providing construction and demolition disposal debris;

processing said construction and demolition disposal debris by separating and recovering a construction and demolition disposal debris recovered screened material product therefrom for use as a substitute daily/initial solid waste disposal landfill cover material, said construction and demolition disposal debris recovered screened material product consisting of particles of which at least 90% have a particle size of 3" or less, wherein said step of processing is selected from the group consisting of:

- (a) shredding by impacting, milling or grinding said construction and demolition disposal debris through 3" openings of a shredder grate surface; and
- (b) screening said construction and demolition disposal debris through 3" openings of a screen surface;

periodically leveling and compacting disposed waste material in the solid waste disposal landfill;

applying a <u>layer</u> of said construction and demolition disposal debris recovered screened material product to the leveled and compacted disposed waste material; and

spraying and soaking said <u>layer</u> of construction and demolition disposal debris recovered screened material product with a solution consisting of:

water;

boric acid;

detergent;

- a nutrient; and
- a deodorant.
- 17. A method as recited in claim 5 further comprising the steps of:

spraying and soaking said recovered screened material product with said solution prior to said step of applying said layer; and

storing said sprayed and soaked recovered screened material product for subsequent use as said substitute daily/initial solid waste landfill cover material.

WEST	
Help Logout Interrupt	
Main Menu Search Form Posting Counts Show S Numbers Edit S Numbers Preferences	Cases
Search Results - Terms Documents 13 not L5 13	
US Patents Full-Text Database US Pre-Grant Publication Full-Text Database JPO Abstracts Database EPO Abstracts Database Derwent World Patents Index IBM Technical Disclosure Bulletins Search: Refine Search Clear	
Search History	

DATE: Tuesday, August 12, 2003 Printable Copy Create Case

Set Name side by side	Query	Hit Count	Set Name result set
•	GPB,JPAB,EPAB,DWPI; PLUR=YES; OP=ADJ		resure see
<u>L6</u>	13 not L5	13	<u>L6</u>
<u>L5</u>	13 and L4	3	<u>L5</u>
<u>L4</u>	granul\$8	367209	<u>L4</u>
<u>L3</u>	11 and L2	16	<u>L3</u>
<u>L2</u>	herbicid\$6 or pesticid\$6	97972	<u>L2</u>
<u>L1</u>	biosolid\$1 or (bio-solid\$1)	307	<u>L1</u>

END OF SEARCH HISTORY

Generate Collection

Print

Search Results - Record(s) 1 through 3 of 3 returned.

☐ 1. Document ID: US 20020098982 A1

L5: Entry 1 of 3

File: PGPB

Jul 25, 2002

PGPUB-DOCUMENT-NUMBER: 20020098982

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020098982 A1

TITLE: Production and use of biosolid granules

PUBLICATION-DATE: July 25, 2002

INVENTOR-INFORMATION:

NAME

CITY

STATE

COUNTRY

RULE-47

Burnham, Jeffrey C.

Naples

FL

US

US-CL-CURRENT: 504/359; 504/117, 504/367, 71/64.02, 71/64.11

Full Title Citation Front Review Classification Date Reference Sequences Attachments Claims KWC Draw, Desc Image

☑ 2. Document ID: US 20020053229 A1

L5: Entry 2 of 3

File: PGPB

May 9, 2002

PGPUB-DOCUMENT-NUMBER: 20020053229

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020053229 A1

TITLE: Organic-based fertilizer

PUBLICATION-DATE: May 9, 2002

INVENTOR-INFORMATION:

NAME

CITY

STATE

COUNTRY

RULE-47

Varshovi, Amir

Gainesville

FL

US

US-CL-CURRENT: 71/6

Full Title Citation Front Review Classification Date Reference Sequences Attachments Claims KMC Draw, Desc Image

3. Document ID: US 20020098982 A1 WO 200257862 A2

L5: Entry 3 of 3

File: DWPI

Jul 25, 2002

DERWENT-ACC-NO: 2002-698524

DERWENT-WEEK: 200316

COPYRIGHT 2003 DERWENT INFORMATION LTD

TITLE: Granular nutrient and micro-organism use for bioremediation and crop

fertilizer has a number of layers and contains biosolid material

INVENTOR: BURNHAM, J C

PRIORITY-DATA: 2001US-272469P (March 2, 2001), 2001US-262631P (January 22, 2001),

2002US-0051387 (January 22, 2002)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
US 20020098982 A1	July 25, 2002		000	A01N025/28
WO 200257862 A2	July 25, 2002	E	025	G05F000/00

 $\text{INT-CL (IPC)} : \underline{\text{A01}} \ \underline{\text{N}} \ \underline{25/00}; \ \underline{\text{A01}} \ \underline{\text{N}} \ \underline{25/12}; \ \underline{\text{A01}} \ \underline{\text{N}} \ \underline{25/28}; \ \underline{\text{A01}} \ \underline{\text{N}} \ \underline{63/00}; \ \underline{\text{G05}} \ \underline{\text{F}} \ \underline{0/00}$

Full Title		Front	Review	Classification	Date	Reference	Sequences	Attachments	KWC
Draw Desc	Image								
•									
				Generate C	`ollecti		Print		
			<u></u>	Generate C	Juliecu		rinit]		
		Tern	ns				Docume	nts	
11		I (111	11.0		·····		Docume		
<u> </u>	nd L4								

Display Format: - Change Format

Previous Page Next Page

Generate Collection Print

L5: Entry 2 of 3

File: PGPB

May 9, 2002

DOCUMENT-IDENTIFIER: US 20020053229 A1

TITLE: Organic-based fertilizer

Abstract Paragraph (1):

The subject invention provides methods for producing homogenous organic base fertilizer for plant nutrition and soil fertility. Also provided by the subject invention are value added fertilizer products for plant nutrition and soil fertility and concentrated liquid formulations/nutrient supplements that provide the value added nutrients to fertilizer products. Methods according to the invention involve the application of concentrated liquid(s) and/or dry formulation(s) comprising a mixture of one or more plant nutrient(s), one or more additional organic compound(s), one or more penetrate(s), and one or more optional supplement(s) into one or more organic base material(s). These organic base materials include, and are not limited to, biosolids, activated sludge, municipal compost, animal manures (e.g., horse, cow, chicken, pig, and sheep), and composted organic byproducts.

Summary of Invention Paragraph (2):

[0002] The continuous use of chemical <u>pesticides</u> and fertilizers on plants, bushes, and trees, and especially in agricultural settings in the production of crops, has created a variety of ecological problems. As the world and national populations have increased, farmers and horticulturalists have come to rely increasingly upon chemically synthesized fertilizers and chemical <u>pesticides</u> to maintain the same level of crop production.

Summary of Invention Paragraph (3):

[0003] More and more, organic base byproducts, including, but not limited to, biosolids, activated sludge, municipal compost, animal manures (such as cow, horse, pig, sheep, or chicken manure), and composted organic byproducts containing various plant nutrients, are being produced as a result of industrial and agricultural activities and processes. The nutrient content of these byproducts is generally low and these byproducts have been used as fillers, soil amendments, or landfill because of their low nutrient value.

Detail Description Paragraph (7):

[0013] Methods according to the invention involve the application of concentrated liquid(s) and/or dry formulation(s) comprising a mixture of one or more plant nutrient(s), one or more additional organic compound(s), and one or more penetrant(s), into one or more organic base material(s). Optionally, one or more beneficial microorganism(s) may be added to the organic base material(s). According to the invention, organic base materials include, and are not limited to, biosolids, activated sludge, municipal compost, animal manures, and composted organic byproducts.

Detail Description Paragraph (8):

[0014] Organic compounds useful in the practice of the subject invention include, and are not limited to, biosolids, humic acid fulvic acid, and plant extracts. Plant extracts include, and are not limited to, seaweed extracts, kelp extracts, and/or the extracts of other plant materials. The subject invention allows for the incorporation of one or more organic compound(s), or combinations thereof, into the value added fertilizer products of the subject invention.

Detail Description Paragraph (9):

[0015] Penetrants useful in the practice of the subject invention include non-ionic wetting agents, detergent based surfactants, silicones, and/or organo-silicones. Non-limiting examples of penetrants include polymeric polyoxyalkylenes, allinol, nonoxynol, octoxynol, oxycastrol, oxysorbic (for example, polyoxyethylated sorbitol fatty-acid esters (TWEEN); thalestol, and polyethylene glycol octylphenol ether (TRITON), Sylgard 309 (straight silicone), Silwet L-77 (straight silicone), Kinetic (silicone/surfactant blend), and Herbex (silicone/surfactant blend). A penetrant can be used for the enhancement of the organic-base byproduct after granulation. In one embodiment, the penetrant is added to the liquid concentrate prior to its incorporation with the granulated organic base byproduct. Alternatively, penetrant can be added, separately, to granulated organic-base byproducts prior to the addition of the liquid concentrate. The use of a penetrant is optional if addition of liquid nutrient concentrate or dry nutrients to the organic-base byproducts occurs prior to granulation.

Detail Description Paragraph (10):

[0016] Organic base materials include, and are not limited to, biosolids, activated sludge, municipal compost, animal manures (e.g., horse, cow, chicken, pig, and/or sheep), and composted organic byproducts. Other organic base materials include processed animal body and vegetable products such as blood meal, feather meal, cottonseed meal, ocean kelp meal, and fish fertilizers such as fish emulsions or meal.

Detail Description Paragraph (12):

[0018] Microorganisms (bacteria, fungi and viruses) that control various types of pathogens in the soil include microorganisms that control soil-born fungal pathogens, such as Trichoderma sp., Bacillus subtilis, Penicillium spp.; microorganisms that control insects, such as Bacillus sp., e.g., Bacillus popalliae; microorganisms that act as herbicides, e.g., Alternaria sp., and the like. These organisms are readily available from public depositories throughout the world.

Detail Description Paragraph (14):

[0020] Optional soil and/or plant additives that can be added to the fertilizer compositions of the invention include water trapping agents, such as zeolites; natural enzymes; growth hormones (such as the gibberellins, including gibberellic acid and gibberellin plant growth hormones); and control agents, including pesticides such as acaracides, molluskicides, insecticides, fungicides, nematocides, and the like.

Detail Description Paragraph (15):

[0021] In one embodiment, liquid nutrients are incorporated into organic base materials by spray application on palletized or <u>granulated</u> organic base materials. Alternatively, the liquid nutrients can be added via direct mixture into the organic base prior to <u>granulation</u>. In either embodiment, value added products for plant nutrition and soil fertility are produced.

Detail Description Paragraph (16):

[0022] In various embodiments, beneficial microorganisms can, optionally, be added to the organic base materials before, during, or after granulation or polarization of the organic base material. In one embodiment, beneficial microbes are added after the granulation or pelletization process.

Detail Description Paragraph (17):

[0023] Concentrated liquid formulation(s) may be applied to an organic base by spraying onto organic base materials as they enter, once they are already in, or as they exit a mixing means. Mixing means include, and are not limited to, rotating mixers, other rotating containers, or granulation drums. The mixing means may rapidly rotate to mix and incorporate the liquid concentrate into the organic base materials. Concentrated liquid formulations or concentrated nutrient supplements are terms that may be used interchangeably throughout the specification.

Detail Description Paragraph (18):

[0024] Dry nutrient formulations may be applied to wet organic base materials prior to the drying and granulation process. The dry formulation(s) react with the wet organic base materials and are mixed through the process of granulation. Dry soluble

macronutrient sources useful in the practice of the subject invention include: nitrogen sources, such as ammonium sulfate; and potassium sources, such as potassium nitrate, potassium phosphate, and potassium sulfate.

Detail Description Paragraph (19):

[0025] In one embodiment, liquid nutrients are incorporated into organic base materials by spray application on pelletized or <u>granulated</u> organic base materials. Alternatively, the liquid nutrients can be added via direct mixture into the organic base prior to <u>granulation</u>. In either embodiment, value added products for plant nutrition and soil fertility are produced.

Detail Description Paragraph (20):

[0026] The concentrated liquid formulation(s) and the dry formulation(s) may vary in regards to type and concentrations of plant nutrients depending on the desired end formulation for the homogenous organic-base granular fertilizer. These formulations may be varied to provide the desired nutritional components for a particular crop, plant, or grass.

Detail Description Paragraph (21):

[0027] Concentrated liquid formulation(s) may be applied to an organic base by spraying onto organic base materials as they enter, once they are already in, or as they exit a mixing means. Mixing means include, and are not limited to, rotating mixers, other rotating containers, or granulation drums. The mixing means may rapidly rotate to mix and incorporate the liquid concentrate into the organic base materials.

Detail Description Paragraph (22):

[0028] Dry nutrient formulations may be applied to wet organic base materials prior to, during, or after the drying and granulation process. The dry formulation(s) react with the wet organic base materials and are mixed through the process of granulation.

Detail Description Paragraph (23):

[0029] The subject invention has been used to enhance <u>biosolids</u> produced by municipalities. The <u>biosolids</u> were enhanced after <u>granulation</u> to produce a higher nutrient value fertilizer and then applied to turfgrass as part of an efficacy study performed at the University of Florida.

Detail Description Paragraph (26):

[0032] A penetrant can be used for the enhancement of the organic-base byproduct after <u>granulation</u>. In one embodiment, the penetrant is added to the liquid concentrate prior to its incorporation with the <u>granulated</u> organic base byproduct. Alternatively, the penetrant can be added, separately, to <u>granulated</u> organic-base byproducts prior to the addition of the liquid concentrate. The use of a penetrant is optional if addition of liquid nutrient concentrate or dry nutrients to the organic-base byproducts occurs prior to <u>granulation</u>. Penetrant is typically used to break the hydrophobic of the <u>granulated</u> organic-base byproduct and to increase absorption of the liquid concentrate.

<u>Detail Description Paragraph</u> (27):

[0033] Liquid nutrient concentrate can be added by spraying, injecting or otherwise incorporating it into the organic-base byproduct after granulation or while the organic-base byproduct is being rotated or mixed in a rotating blender/mixer or other apparatus. The liquid concentrate should be added uniformly so as to allow for uniform absorption by the organic-base byproduct granules. In some embodiments, the addition of the liquid concentrate should be performed incrementally to allow time for absorption by the organic-base byproduct granules. The amount added incrementally depends on the amount of organic-base byproducts being enhanced and the end formulation. Fifteen to thirty minute intervals can be provided between each incremental addition depending on the total amount to be added, the moisture content of the organic-base byproducts at the different stages of enhancement, and the ambient temperature.

Detail Description Paragraph (28):

[0034] When utilizing the method of this invention for preparation of slow release

homogenous organic-base fertilizer, the liquid concentrate added to the organic-base byproduct should be between 1% to 50%, preferably 1.5% to 40%, more preferably 2% to 30%, and most preferably between 2.5% to 25% of the total weight of the finished product depending on the desired formulation. When the liquid concentrate is added after granulization of the organic-base byproduct, the following macronutrient sources are preferred for enhancing the nutrient value of the organic-base byproduct: nitrogen (N) sources, including urea, ammonium nitrate, ammonium sulfate, methylane urea, and/or urea formaldehyde; potassium (K) sources, including potassium nitrate, potassium thiosulfate, potassium phosphate, potassium hydroxide, potassium sulfate, and/or potassium carbonate; and phosphorus (P) sources, including ammonium phosphate, potassium phosphate, and/or phosphoric acid. Any combination of these macronutrients may be used in the practice of the invention and one or more of the N, P, or K sources may be used in formulating compositions according to the subject invention.

Detail Description Paragraph (30):

[0036] High quality homogenous slow-release organic-base fertilizer can be prepared prior to drying and granulation of the organic-base byproduct(s) by incorporating selected dry soluble plant nutrients and additional organic substrates into organic-base byproducts containing 70% to 90% moisture. If this method is used, the selection of added materials must be based on their solubility and their stability at temperatures of about 85.degree. to 100.degree. C. The solubility of selected plant nutrients should be high in organic-base byproducts containing 70% to 90% moisture. The dry soluble plant nutrients and organic substrates are incorporated into the organic-base byproducts in a mixing/homogenizer chamber or auger prior to the dryer/granulator.

Detail Description Paragraph (35):

[0039] Fertilizers produced according to the subject invention were evaluated to compare the quality, growth and N uptake response of three slow-release N sources. On a comparative basis, the Green Technologies, Inc. granular slow-release fertilizer (GT-6-2-4) produced higher visual quality, growth rate, and total N uptake during two 56-day evaluation cycles than did MILORGANITE or NITROFORM.

CLAIMS:

- 2. The method of claim 1, wherein a dry soluble plant nutrient, and/or organic compounds or a combination of dry soluble plant nutrients and/or organic compounds is added and mixed with the organic-base byproducts containing 70% to 90% moisture, prior to their entry into a rotary_granulator/dryer.
- 4. The method of claim 1, wherein said fertilizer is mixed in a rotary granulator/dryer.
- 5. The method of claim 1, wherein said liquid concentrate is added to the organic-base byproduct prior to their entry into the rotary granulator/dryer.
- 6. The method of claim 1, wherein said liquid concentrate is added to the organic-base byproduct when said organic-base byproduct is in said rotary granulator/dryer.
- 7. The method of claim 1, wherein said liquid concentrate is added to said organic base byproduct after said organic base byproduct exits said rotary granulator/dryer.
- 8. The method of claim 1, wherein said liquid concentrate is added to said organic base byproduct as it enters said rotary granulator/dryer.
- 9. The method of claim 1, wherein said liquid concentrate is added to said organic base byproduct in said rotary granulator/dryer.
- 11. The method of claim 1, wherein said plant nutrient can be nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), iron (Fe), zinc (Zn), manganese (Mn), copper (Cu), and boron (B), or any combination thereof; said dry soluble plant nutrient comprises ammonium sulfate, potassium nitrate, potassium

phosphate, potassium sulfate, or any combination thereof; said beneficial microbe can be bacterial, fungal, viral, or any combination thereof; said organic compound can be biosolids, humic acid, fulvic acid, plant extract, seaweed extract, kelp extract, extracts of other plant materials, or any combination thereof; said penetrant comprises one or more non-ionic surfactant(s), one or more wetting agent(s), one or more detergent based surfactant(s), one or more silicone(s), and/or one or more organo-silicone(s) or any combination thereof; said other soil and plant additives comprise water trapping agents, zeolites, natural enzymes, growth hormones, gibberellins, gibberellic acid, and weed and/or pest control agents, acaracides, molluskicides, insecticides, fungicides, nematocides, or any combinations thereof; and one or more organic base byproduct(s) selected from the group consisting of biosolids, activated sludge, municipal compost, animal manures, composted organic byproducts, processed animal body and vegetable products, blood meal, feather meal, cottonseed meal, ocean kelp meal, fish fertilizers, fish emulsions, and fish meal.

16. The high quality homogenous slow-release organic-base fertilizer composition of claim 13, wherein said plant nutrient can be nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), iron (Fe), zinc (Zn), manganese (Mn), copper (Cu), and boron (B), or any combination thereof; said dry soluble plant nutrient comprises ammonium sulfate, potassium nitrate, potassium phosphate, potassium sulfate, or any combination thereof; said beneficial microbe can be bacterial, fungal, viral, or any combination thereof; said organic compound can be biosolids, humic acid, fulvic acid, plant extract, seaweed extract, kelp extract, extracts of other plant materials, or any combination thereof; said penetrant comprises one or more non-ionic surfactant(s), one or more wetting agent(s), one or more detergent based surfactant(s), one or more silicone(s), and/or one or more organo-silicone(s), or any combination thereof; said other soil and plant additives comprising water trapping agents, zeolites, natural enzymes, growth hormones, gibberellins, gibberellic acid, and weed and/or pest control agents, ascaracides, molluskicides, insecticides, fungicides, nematocides, or any combinations thereof; and one or more organic-base byproduct(s) selected from the group consisting of biosolids, activated sludge, municipal compost, animal manures, composted organic byproducts, processed animal body and vegetable products, blood meal, feather meal, cottonseed meal, ocean kelp meal, fish fertilizers, fish emulsions, and fish meal.

WEST

Generate Collection

Print

Search Results - Record(s) 1 through 10 of 13 returned.

☐ 1. Document ID: US 20030146169 A1

L6: Entry 1 of 13

File: PGPB

Aug 7, 2003

PGPUB-DOCUMENT-NUMBER: 20030146169

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030146169 A1

TITLE: Methods of synthesizing an oxidant and applications thereof

PUBLICATION-DATE: August 7, 2003

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47 Ciampi, Lee Edward Belle Isle FL US

Smith, Gregory F. Elyria OH US
Knoble, Bernie Avon OH US

US-CL-CURRENT: 210/716; 210/192

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

KMC

☐ 2. Document ID: US 20030087422 A1

L6: Entry 2 of 13

File: PGPB

May 8, 2003

PGPUB-DOCUMENT-NUMBER: 20030087422

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030087422 A1

TITLE: Biogenic FPA: enzymatic production of formylphosphonic acid by the oxidation

of hydroxymethylphosphonic acid

PUBLICATION-DATE: May 8, 2003

INVENTOR - INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Heitkamp, Michael A. Aiken SC US Hughes, B. Mason St. Louis MO US Viswanath, Shekhar St.Louis MO US

US-CL-CURRENT: 435/262.5; 435/252.3

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw, D	esc Ir	nage							

KOMC

☐ 3. Document ID: US 20030080224 A1

L6: Entry 3 of 13

File: PGPB

May 1, 2003

PGPUB-DOCUMENT-NUMBER: 20030080224

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030080224 A1

TITLE: Two-stage comminuting and dehydrating system and method

PUBLICATION-DATE: May 1, 2003

INVENTOR - INFORMATION:

NAME

CITY

STATE

RULE-47

Rowley, Frank F. JR.

Valley Center

KS

US

COUNTRY

US-CL-CURRENT: 241/5; 241/152.1, 241/29, 241/39

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

4. Document ID: US 20030031511 A1

L6: Entry 4 of 13

File: PGPB

Feb 13, 2003

PGPUB-DOCUMENT-NUMBER: 20030031511

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030031511 A1

TITLE: Devices, systems, and methods for controlling erosion

PUBLICATION-DATE: February 13, 2003

INVENTOR-INFORMATION:

NAME

CITY

STATE

COUNTRY

RULE-47

Tyler, Rodney W.

Medina

OH

US

US-CL-CURRENT: 405/15; 405/107, 405/302.6, 405/36

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Drawl Description

KWIC

5. Document ID: US 20020155044 A1

L6: Entry 5 of 13

File: PGPB

Oct 24, 2002

PGPUB-DOCUMENT-NUMBER: 20020155044

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020155044 A1

TITLE: Methods of synthesizing an .oxidant and applications thereof

PUBLICATION-DATE: October 24, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Ciampi, Lee Edward Belle Isle FL US Smith, Gregory F. Elyria OH US Knoble, Bernie Avon OH US

US-CL-CURRENT: 422/187; 422/188, 422/198, 423/632

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC Draw, Desc Image

☐ 6. Document ID: US 20020121482 A1

L6: Entry 6 of 13 File: PGPB Sep 5, 2002

PGPUB-DOCUMENT-NUMBER: 20020121482

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020121482 A1

TITLE: Methods of synthesizing an oxidant and applications thereof

PUBLICATION-DATE: September 5, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Ciampi, Lee Edward Belle Isle FL US Smith, Gregory F. Elyria OH US Knoble, Bernie Avon OH US

US-CL-CURRENT: 210/716; 210/758, 423/594.2

7. Document ID: US 20020069685 A1

L6: Entry 7 of 13 File: PGPB Jun 13, 2002

PGPUB-DOCUMENT-NUMBER: 20020069685

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020069685 A1

TITLE: Recycled soil

PUBLICATION-DATE: June 13, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Adam, Paul Thomas Flinton PA US

US-CL-CURRENT: 71/11

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC

Draw. Desc Image

☐ 8. Document ID: US 20020000485 A1

L6: Entry 8 of 13

File: PGPB

Jan 3, 2002

PGPUB-DOCUMENT-NUMBER: 20020000485

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020000485 A1

TITLE: Two-stage comminuting and dehydrating system and method

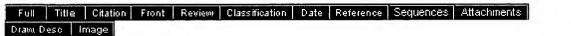
PUBLICATION-DATE: January 3, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Rowley, Frank F. JR. Valley Center KS US

US-CL-CURRENT: 241/39



KWIC

9. Document ID: US 6517015 B2

L6: Entry 9 of 13

File: USPT

Feb 11, 2003

US-PAT-NO: 6517015

DOCUMENT-IDENTIFIER: US 6517015 B2

TITLE: Two-stage comminuting and dehydrating system and method

DATE-ISSUED: February 11, 2003

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Rowley, Jr.; Frank F. Valley Center KS 67147

US-CL-CURRENT: 241/5; 241/152.1, 241/29, 241/33, 241/39

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

☐ 10. Document ID: US 6419722 B1

L6: Entry 10 of 13 File: USPT Jul 16, 2002

US-PAT-NO: 6419722

DOCUMENT-IDENTIFIER: US 6419722 B1

TITLE: Recycled soil

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Adam; Paul Thomas

Flinton

PA

16640

 $\text{US-CL-CURRENT: } \underline{71/13}; \ \underline{47/4}, \ \underline{71/15}, \ \underline{71/24}, \ \underline{71/25}, \ \underline{71/903}, \ \underline{71/904}$

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC	
Draw, D	eso Ima	age									
											
					Generate C	ollecti	on	Print			
	[·····				
	Terms						Documents				
	13 not	L5								13	

Display Format: -

Change Format

Previous Page

Next Page

Generate Collection

Print

Search Results - Record(s) 11 through 13 of 13 returned.

☐ 11. Document ID: US 6399359 B1

L6: Entry 11 of 13

File: USPT

Jun 4, 2002

US-PAT-NO: 6399359

DOCUMENT-IDENTIFIER: US 6399359 B1

TITLE: Composition system

DATE-ISSUED: June 4, 2002

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Hofstede; Harrie West Leederville. W.A. 6007

US-CL-CURRENT: 435/262.5; 435/290.1, 435/290.4, 71/9

Full Title Citation Front Review Classification Date Reference Sequences Attachments Draw Desc Image

KMC

ΑU

☐ 12. Document ID: US 6281001 B1

L6: Entry 12 of 13

File: USPT

Aug 28, 2001

US-PAT-NO: 6281001

DOCUMENT-IDENTIFIER: US 6281001 B1

TITLE: Process for controlled composting of organic material and for bioremediating

soils

DATE-ISSUED: August 28, 2001

INVENTOR-INFORMATION:

ZIP CODE COUNTRY CITY STATE NAME

McNelly; James J. St. Cloud MN56303

US-CL-CURRENT: $\frac{435}{262}$; $\frac{210}{612}$, $\frac{210}{613}$, $\frac{210}{620}$, $\frac{435}{262.5}$, $\frac{435}{290.1}$, $\frac{71}{8}$, $\frac{71}{9}$, <u>71/903</u>

Full Title Citation Front Review Classification Date Reference Sequences Attachments Draw, Desc Image

☐ 13. Document ID: US 5958241 A

L6: Entry 13 of 13

File: USPT

Sep 28, 1999

KWIC

US-PAT-NO: 5958241

DOCUMENT-IDENTIFIER: US 5958241 A

** See image for Certificate of Correction **

TITLE: Waste treatment and minimization system

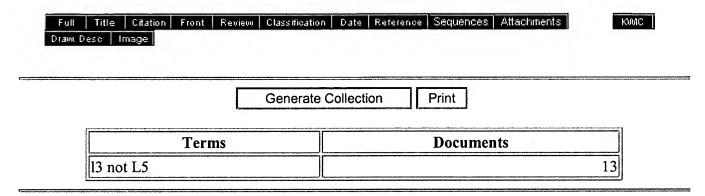
DATE-ISSUED: September 28, 1999

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

DeBenedetto; Ronald Peter Wilmer AL Leftwich; Eugene Porter Mobile AL

US-CL-CURRENT: 210/611; 210/620, 210/626



Display Format: - Change Format

Previous Page Next Page

Generate Collection Print

L6: Entry 12 of 13

File: USPT

Aug 28, 2001

DOCUMENT-IDENTIFIER: US 6281001 B1

TITLE: Process for controlled composting of organic material and for bioremediating soils

Brief Summary Text (4):

Landfill space is dwindling, industrial and municipal waste production is increasing, and the disposal of solids from waste water treatment is becoming more complex. Composting is a viable alternative for disposal of organic materials from nearly all waste streams. Composting can be used to process municipal waste water biosolids, to remediate industrial waste water biosolids, and to treat wastes from processing food and agricultural products. Composting can reduce the volume of organic waste materials by about 50% or more while producing a stable non-odorous material that can be used as fertilizer or an amendment for soil.

Detailed Description Text (48):

The organic material can be sludge, such as municipal waste water treatment residuals or industrial waste water treatment residuals. The organic material can be biosolids, which is waste water residual from a publicly owned waste water treatment plant. Another type of organic material is waste and byproducts from processing crops, foods, and other agricultural products. Animal processing waste and byproducts, such as entrails or carcasses, are organic material that can be composted. Manufactured or industrial products can be compostable organic material as well. Compostable manufactured or industrial products include manufactured cellulose products, explosive materials, petroleum products, agricultural chemicals, and the like.

Detailed Description Text (49):

The composting method is advantageous for composting biosolids. Biosolids are an odorous composition that can be produced in large volume, and which must be thickened and managed to prevent production of annoying odors. Traditional waste water treatment processes are only marginally effective for reducing volume and odor. Typical processing by mechanical thickening and concentration by the use of clarifiers and flocculating agents produces effluent that is only about 1% solids. This mixture can be processed further through a belt press or a centrifuge or by addition of polymers to yield biosolids. Conventional processing can yield as biosolids an organic material that includes water and about 12 percent by weight to about 22 percent by weight solids. Typically the biosolid organic material from municipal waste water treatment will be about 15 percent by weight to about 20 percent by weight solids. The composition of such biosolids can be adjusted to make it compatible with the composting process.

Detailed Description Text (51):

Continuous or intermittent evaluating and adjusting the composition of organic material can allow composting of toxic or otherwise incompatible material. The process of composting toxic or otherwise incompatible organic materials is called "remediation" or "bioremediation". Toxic or incompatible organic materials that can be remediated include hydrocarbons and synthetic organic compounds such as hetricomposition/ oil, petroleum products, diesel fuel, gasoline, hydraulic fluid, obsolete ordinance, and TNT. Soil including about 2% oil, other petroleum distillates or synthetic organic chemicals can be readily composted by mixing with other organic material. Biosolids is a preferred organic material for adjusting the content of toxic or incompatible organic material. For example, once the presence of

such incompatible materials is known or suspected, their amount can be kept to compatible low levels or they can be mixed with a compatible material. Preferably in remediation, the soil or other matrix contaminated with the incompatible organic material is mixed with organic material that is already at least partly composted.

Detailed Description Text (63):

Typical organic materials are low in carbon and have a low carbon:nitrogen ratio. For example, biosolids are typically about 6 percent by weight nitrogen, which results in a low carbon:nitrogen ratio of about 12:1. The carbon:nitrogen ratio is advantageously increased by adding a material with high levels of carbon. Amendments useful for increasing the carbon:nitrogen ratio of an organic material include paper, sawdust, leaves, straw, older compost, animal manure, a bulking agent, and mixtures of these materials.

Detailed Description Text (77):

The amount of inoculant is chosen based on the composition of the organic material or the compostable mixture. For different compostable mixtures and different organic materials the amount of inoculant is adjusted to result in conversion of the organic materials to composted organic materials within an acceptable time, typically more than about 3 days but less than about two or three weeks. The amount of inoculant added is seldom less than about 5 percent by weight of the total compostable mixture. When the organic material includes substances that are toxic to the microbes, that are incompatible with the composting process, or that are particularly difficult for the microbes to break down, the level of the inoculant can be increased to as much as 50% of the compostable mixture. High levels of inoculant allow effective composting in less than optimal conditions. Such high levels of inoculant are typical of and preferable in composting processes used for remediation. More typically, such as when the organic material is routine biosolids, the inoculant will make up about 5 percent by weight to about 20 percent by weight of the compostable mixture. Preferably, for routine organic material the inoculant makes up about 10 percent by weight to about 15 percent by weight of the compostable mixture. More preferably the amount of inoculant is about 10 percent by weight of the compostable mixture.

Detailed Description Text (78):

Typically, inoculant includes previously composted organic material. The old compost is used as a seed culture and contains active organisms that are mixed with organic material, amendment, and bulking agent to form a compostable mixture. Preferred inoculants include composted or composting biosolids, bulking agent recovered from composted or composting biosolids, aged and cured compost, compost from recent batches, microbes cultured for inoculating in various stages of decomposition, cultured and isolated microbes, and mixtures of these inoculants.

Detailed Description Text (84):

Incubation is carried out for sufficient time to result in effective composting of the organic material although other durations can also be used. Typically, an incubation period is 3, 7, 14, or 21 days in total for a batch of organic material. In some applications, the incubation for composting can be supplemented by retention in the sealable container for pathogen destruction. Pathogen destruction can be accomplished by 72 continuous hours of incubation at more than 55 degree. C. under conditions described as "in-vessel composting" under the Rules for Biosolids Management of the United States Environmental Protection Agency, Part 503.

Detailed Description Text (91):

Another advantage of this composting process of the present invention is that maintaining controlled incubation conditions result in rapid and complete composting of organic materials. For example, about 40,000 pounds of biosolids can typically be composted in a 40 cubic yard container in less than about 3 weeks. Remediation of about 30,000 pounds of soil contaminated with petroleum products or agricultural chemicals can be complete in a 40 cubic yard container in less than about 3 weeks. Proper choice of incubation conditions and monitoring to maintain these conditions facilitates rapid composting.

WEST

End of Result Set

Generate Collection Print

L6: Entry 13 of 13

File: USPT

Sep 28, 1999

DOCUMENT-IDENTIFIER: US 5958241 A

** See image for Certificate of Correction **
TITLE: Waste treatment and minimization system

Brief Summary Text (4):

DAF, API Separator and similar treatment units may also be found in other industrial facilities, such as those producing or utilizing chemicals, petrochemicals, polymers, rubbers, pesticides, oil and gas, hydrocarbon, synthetics or organics. Wastes and discharges from these types of facilities could be listed or otherwise characteristically hazardous, if not listed, and thus likely candidates for waste minimization utilizing the invention disclosed herein.

Brief Summary Text (25):

The water application of the invention applies to a plant's regular biological wastewater treatment system. Under current normal operating methods, biosolids, often called waste activated sludge (WAS) are routinely removed from a bioreactor (hereinafter referred to as the water bioreactor) in order to maintain the proper food to mass ratio (contaminates to microorganisms) or F/M ratio. This typically translates into a volatile suspended solids (VSS) content in the water bioreactor of 2,000 to 4,000 mg/L. In order to maintain this concentration level, WAS must be removed. The wasted sludge is then transferred to some sort of final resting place, i.e. lagoon, sludge pond, digester, etc. and eventually requires dewatering and disposal, either on land or in landfills. Land application is becoming more difficult because of increased regulation.

Brief Summary Text (29):

In carrying out the method of, or in operating the system of, the oil and grease application of the invention, wastewater sludges are routed to the oil and grease bioreactor. Material in the oil and grease bioreactor, consisting of biomass solids, sludge and liquids, flows to a liquids/solids separator wherein the rate of such outflow from the oil and grease bioreactor is essentially equal to the combined rate of inflow to the oil and grease bioreactor of (a) the aforesaid wastewater sludges and (b) concentrated biosolids produced in the liquid/solids separator. Separated liquids produced by the liquid/solid separator are returned to the inlet of the existing wastewater system. The wastewater proceeds for routine treatment (e.g., through the water bioreactor) prior to discharge. The wastewater treatment process continues and additional sludges from the wastewater system are introduced into the oil and grease bioreactor.

Brief Summary Text (30):

Concentrated biosolids produced in the liquid/solids separator are returned to the oil and grease bioreactor for further treatment. Therefore, no solids are removed from the oil and grease bioreactor or any part of the essentially closed-loop system during normal operating conditions.

Set Name side by side		Hit Count	Set Name result set
DB=U	SPT,PGPB,JPAB,EPAB,DWPI; PLUR=YES; OP=ADJ		
<u>L21</u>	14 and L20	9	<u>L21</u>
<u>L20</u>	L2 and 113	125	<u>L20</u>
<u>L19</u>	(l11 and L17) not l18	24	<u>L19</u>
<u>L18</u>	ll1 same L17	1	<u>L18</u>
<u>L17</u>	microorganism\$1 or bacteri\$ or microbiolog\$	335046	<u>L17</u>
<u>L16</u>	111 and L15	0	<u>L16</u>
<u>L15</u>	bioremediat\$5	1464	<u>L15</u>
<u>L14</u>	111 and L13	0	<u>L14</u>
<u>L13</u>	bioremediat\$5 same (microorganism\$1 or bacteri\$ or microbiolog\$)	773	<u>L13</u>
<u>L12</u>	17 and L11	10	<u>L12</u>
<u>L11</u>	l5 same L10	61	<u>L11</u>
<u>L10</u>	L9 with 13	1466	<u>L10</u>
<u>L9</u>	multilayer\$3 or ((layer\$3 or coat\$4) near4 (several or many or multiple or multi))	364625	<u>L9</u>
<u>L8</u>	l6 and L7	42	<u>L8</u>
<u>L7</u>	fertili\$ or micronutrient\$1	84281	<u>L7</u>
<u>L6</u>	l4 same L5	229	<u>L6</u>
. <u>L5</u>	L2 with 13	2930	<u>L5</u>
<u>L4</u>	11 with L3	22198	<u>L4</u>
<u>L3</u>	granul\$6	347479	<u>L3</u>
<u>L2</u>	(delay\$3 or control\$4 or time\$1) near3 (releas\$3 or deliver\$4)	248881	<u>L2</u>
<u>L1</u>	multilayer\$3 or layer\$3	2784790	<u>L1</u>

END OF SEARCH HISTORY

WEST

Generate Collection

Print

Search Results - Record(s) 1 through 10 of 10 returned.

☐ 1. Document ID: US 20020024166 A1

L12: Entry 1 of 10

File: PGPB

Feb 28, 2002

PGPUB-DOCUMENT-NUMBER: 20020024166

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020024166 A1

TITLE: Punch and die

PUBLICATION-DATE: February 28, 2002

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Fukada, Hiroshi Toyonaka-shi JP Matoba, Hiroshi Osaka-shi JP Nakamura, Etsuji Osaka-shi JP

US-CL-CURRENT: 264/123; 425/352, 425/470

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMIC
Draw, D	esc l	mage									

☑ 2. Document ID: US 6039781 A

L12: Entry 2 of 10

File: USPT

Mar 21, 2000

US-PAT-NO: 6039781

DOCUMENT-IDENTIFIER: US 6039781 A

TITLE: Precoated controlled release <u>fertilizers</u> and processes for their preparation

DATE-ISSUED: March 21, 2000

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Goertz; Harvey M. Marysville OH
Timmons; Richard J. Marysville OH
Johnson; William R. San Jose CA

US-CL-CURRENT: 71/1; 71/28, 71/64.01, 71/64.07, 71/64.11

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC
Draw, D	esc Ir	nage									

Ç,

☑ 3. Document ID: US 5849060 A

L12: Entry 3 of 10

File: USPT

Dec 15, 1998

US-PAT-NO: 5849060

DOCUMENT-IDENTIFIER: US 5849060 A

TITLE: Controlled release fertilizer and preparations thereof

DATE-ISSUED: December 15, 1998

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY
Diping; Li Zhengzhou CN
Haobin; Wang Zhengzhou CN

Haobin; Wang Zhengzhou CN
Xiucheng; Xu Zhengzhou CN
Cuihong; Hou Zhengzhou CN

US-CL-CURRENT: 71/64.07; 71/28, 71/29, 71/30, 71/31, 71/32, 71/33, 71/34, 71/35, 71/36, 71/64.11

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC
Draw. D	eso Ir	nage									

☑ 4. Document ID: US 5567221 A

L12: Entry 4 of 10

File: USPT

Oct 22, 1996

US-PAT-NO: 5567221

DOCUMENT-IDENTIFIER: US 5567221 A

TITLE: Compositions and methods for use in aquaculture

DATE-ISSUED: October 22, 1996

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Larson; Steven D. Houston TX Kastner; Richard J. Gulfport MI

US-CL-CURRENT: 71/28; 119/212, 119/230, 47/1.4, 71/64.07, 71/64.11, 71/64.13

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draws Description

☐ 5. Document ID: US 4842790 A

L12: Entry 5 of 10

File: USPT

Jun 27, 1989

US-PAT-NO: 4842790

DOCUMENT-IDENTIFIER: US 4842790 A

TITLE: Method and apparatus for producing high-strength grannular particulates from low-strength prills

DATE-ISSUED: June 27, 1989

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Nunnelly; Luther M. Florence AL

US-CL-CURRENT: 264/117; 118/303, 264/37.29, 425/222, 427/212, 427/242, 71/64.02

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

6. Document ID: US 4019890 A

L12: Entry 6 of 10

File: USPT

Apr 26, 1977

US-PAT-NO: 4019890

DOCUMENT-IDENTIFIER: US 4019890 A

TITLE: Method for producing coated fertilizer

DATE-ISSUED: April 26, 1977

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY Fujita; Toshio Fujishi JA Takahashi; Chigo Fujishi JA Ohshima; Masanari Fujishi JA Ushioda; Tsunezo Tokyoto JA Shimizu; Hirozo Fujishi JA

US-CL-CURRENT: 71/64.11; 427/212, 71/35

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

☐ 7. Document ID: US 3991225 A

L12: Entry 7 of 10

File: USPT

Nov 9, 1976

US-PAT-NO: 3991225

DOCUMENT-IDENTIFIER: US 3991225 A

TITLE: Method for applying coatings to solid particles

DATE-ISSUED: November 9, 1976

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Blouin; Glenn M. Florence AL

US-CL-CURRENT: $\frac{427}{215}$; $\frac{23}{293A}$, $\frac{23}{293S}$, $\frac{23}{313FB}$, $\frac{427}{2.19}$, $\frac{427}{212}$, $\frac{427}{212}$, $\frac{427}{214}$, $\frac{427}{220}$, $\frac{427}{221}$, $\frac{427}{242}$, $\frac{71}{64.02}$, $\frac{71}{64.07}$

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

☐ 8. Document ID: US 3877415 A

L12: Entry 8 of 10

File: USPT

Apr 15, 1975

US-PAT-NO: 3877415

DOCUMENT-IDENTIFIER: US 3877415 A

TITLE: Apparatus for applying coatings to solid particles

DATE-ISSUED: April 15, 1975

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Blouin; Glenn M.

Florence

AL

US-CL-CURRENT: 118/303; 118/314, 118/315, 118/DIG.2, 427/424

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw, D	esc Ir	nage							- 3

KMAC

9. Document ID: DE 4127459 A1 HU 218166 B WO 9304017 A1 AU 9224399 A DE 4127459 C2 ZA 9206284 A PT 100787 A NO 9400386 A FI 9400776 A EP 599927 A1 CZ 9400367 A3 AU 652663 B JP 07500560 W NZ 244013 A HU 70289 T IL 102868 A EP 599927 B1 DE 59208613 G ES 2104939 T3 CZ 282958 B6 RU 2091357 C1 NO 303908 B1

L12: Entry 9 of 10

File: DWPI

Feb 25, 1993

DERWENT-ACC-NO: 1993-067839

DERWENT-WEEK: 200039

COPYRIGHT 2003 DERWENT INFORMATION LTD

TITLE: Fertiliser granules coated with several distinct layers of polyurethane - formed by hardening poly:isocyanate-poly:ol coating mixt., provide sustained, diffusion controlled nutrient release

INVENTOR: BURGER, H; JASCHKOWITZ, M; KLOTH, B; KOHL, W; WEGENER, H; WEHR, P; BUERGER, H

PRIORITY-DATA: 1991DE-4127459 (August 20, 1991)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
DE 4127459 A1	February 25, 1993		011	C05G003/00
HU 218166 B	June 28, 2000		000	C05G003/00
WO 9304017 A1	March 4, 1993	G	022	C05G003/00
AU 9224399 A	March 16, 1993		000	C05G003/00
DE 4127459 C2	July 29, 1993		011	C05G003/00
ZA 9206284 A	July 28, 1993		019	C05G000/00
PT 100787 A	September 30, 1993		000	C05G005/00
NO 9400386 A	February 7, 1994		000	C05G003/00
FI 9400776 A	February 18, 1994		000	C05G000/00
EP 599927 A1	June 8, 1994	G	000	C05G003/00
CZ 9400367 A3	July 13, 1994		000	C05G003/00
AU 652663 B	September 1, 1994		000	C05G005/00
JP 07500560 W	January 19, 1995		000	C05G003/00
NZ 244013 A	February 24, 1995		000	C05G005/00
HU 70289 T	September 28, 1995		000	C05G003/00
IL 102868 A	October 31, 1995		000	C05G003/00
EP 599927 B1	June 11, 1997	G	011	C05G003/00
DE 59208613 G	July 17, 1997		000	C05G003/00
ES 2104939 T3	October 16, 1997		000	C05G003/00
CZ 282958 B6	November 12, 1997		000	C05G003/00
RU 2091357 C1	September 27, 1997		800	C05G003/00
NO 303908 B1	September 21, 1998		000	C05G003/00

599927 B1 INT-CL (IPC): B29B 9/16; C05G 0/00; C05G 3/00; C05G 3/10; C05G 5/00

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Draw. D			TIOHE	MEDIEOD	Classification	Marie	Meterelice	Seddelless	Attaciments	I.

☑ 10. Document ID: US 3259482 A

L12: Entry 10 of 10

DERWENT-ACC-NO: 1968-63337P

DERWENT-WEEK: 196800

COPYRIGHT 2003 DERWENT INFORMATION LTD

TITLE: Slow release fertiliser coated with resin comprising:- (a) a core of granular

fertiliser (at least one soluble nutrient contg. N, P or K); (b) a num

PRIORITY-DATA: 1965US-0496135 (October 14, 1965), 1961US-0132383 (August 18, 1961)

PATENT-FAMILY:

PUB-NO PUB-DATE LANGUAGE PAGES MAIN-IPC

US 3259482 A 000



Generate Collection

Print

File: DWPI

Generate Collection Print

L12: Entry 2 of 10 File: USPT Mar 21, 2000

DOCUMENT-IDENTIFIER: US 6039781 A

TITLE: Precoated controlled release fertilizers and processes for their preparation

Abstract Text (1):

A controlled release <u>fertilizer</u> formed from nutrient granules having intermediate or precoat layers of organic oil, such as raw linseed oil, prior to application of a polymeric, encapsulating coat. The precoat may also contain a binding agent, such as a fine clay, and a drier. The new <u>fertilizer</u> composition is made by a process of applying the oil precoat, but not <u>curing</u> it until after the encapsulating coat is applied. The use of the precoat provides more desirable controlled (delayed) release characteristics at a lower weight of the encapsulating coat, particularly for nutrient cores having a relatively large number of surface imperfections or discontinuities.

Brief Summary Text (3):

This invention relates to controlled release <u>fertilizers</u> and processes for their preparation. More particularly, it relates to <u>particulate fertilizer</u> compositions formed from nutrient granules (such as NPK granules) having <u>intermediate layers</u> or precoats and encapsulating or outer coats applied thereon. It also relates to processes for producing particulate <u>fertilizer</u> compositions that have desirable controlled release characteristics.

Brief Summary Text (5):

Fertilizers have been used for thousands of years to supplement nutrients in growing media. It has been known for some time that the benefit provided by the additional nutrient may depend on when it is delivered to the growing media and, in turn, made available to plants growing in the growing media. Sudden delivery of too much fertilizer can be wasteful or even detrimental to the plants. Delivery of too little fertilizer or delayed delivery of an adequate amount, on the other hand, may starve plants. It is desirable to provide particulate fertilizer compositions that deliver a relatively uniform rate of nutrient to the growing media over time or another specific release pattern.

Brief Summary Text (6):

Delivery of the correct amount of nutrient during an extended growing period previously required multiple applications of a relatively small amount of <u>fertilizer</u> compositions, a very labor intensive method. Accordingly, controlled release <u>fertilizers</u> were developed. Currently, it is desirable to apply the <u>fertilizer</u> once every few weeks to several months.

Brief Summary Text (7):

Generally, controlled release <u>fertilizers</u> used coatings around the nutrient granule to act as a physical or chemical barrier between the nutrient core and the ambient growing media The barrier delayed contact of the nutrient core with moisture and thereby delayed the moisture dissolving the core, and release of the nutrient into the growing media The rate of release of nutrient depended on the material used as the barrier, and its thickness and integrity, among other factors.

Brief Summary Text (8):

One approach to utilizing the barrier or encapsulating technique is shown in U.S. Pat. No. 3,223,518, issued Dec. 14, 1965 to Louis I. Hansen and assigned on its face to Archer-Daniels-Midland. The Hansen patent contemplates <u>fertilizer</u> products having

single or multiple layers of a primer coating applied onto a nutrient core with single or multiple encapsulating layers. As described in the Hansen patent, the primer coating was fully cured before application of the encapsulating coating. The resulting <u>fertilizer</u> showed delayed release of the core nutrient as compared to a completely uncoated nutrient granule. However, repeated application of coatings and curing thereof has been found to be time consuming and commercially unfeasible. Furthermore, <u>fertilizers</u> made according to the teachings of Hansen, having only one primer and only one encapsulating coating, are disclosed by Hansen as releasing from 30-40% of the nutrient within 6 hours of contact with moisture and 50-60% within 24 hours. Such a product delivers too much nutrient too quickly to be acceptable for some controlled release fertilizer applications.

Brief Summary Text (10):

A recognized shortcoming of the application of a polymeric barrier, such as the OSMOCOTE.RTM. encapsulating layer, to a nutrient core was that the release properties of the <u>fertilizer</u> was dependent on the quality of the core or substrate over which the polymer was applied. Discontinuities in the surface of the substrates, such as holes in prilled substrates, deformed granules, or particles having cracks, crevices or irregularities, created incomplete or non-uniform coverage by the coating. Typically for lower coating weights, such as 5 parts per hundred of the core weight ("PPH"), not enough encapsulating material is provided to adequately cover the defects in the core surface of low quality core granules and an unacceptable amount of imperfectly coated particles are created. Predictably, too many of the resulting particles in a <u>fertilizer</u> composition release too much nutrient in the first few days, making them inappropriate for some controlled release (delayed release) products.

Brief Summary Text (13):

It is, therefore, a primary object of the invention to provide a <u>fertilizer</u> in which the nutrient core bears a preliminary or intermediate coating to <u>enhance</u> encapsulation by subsequent outer coats.

Brief Summary Text (14):

It is another object of the invention to provide a <u>fertilizer</u> in which desirable release characteristics are obtained, particularly for nutrient cores with a high degree of surface imperfections.

Brief Summary Text (15):

A further and related significant object is to provide a <u>fertilizer</u> in which the nutrient core <u>granules</u> bear <u>multilayer</u> coatings which are cured after application to the core to allow for desirable <u>controlled release</u> patterns that otherwise might not be obtainable, with or without use of higher coating weights.

Brief Summary Text (16):

Another important object is to provide a <u>fertilizer</u> in which imperfections on the surface of the nutrient granules are covered and/or filled with the use of relatively lower coating weights of the encapsulating layer to avoid thick layers that are expensive and cause lock off of nutrient release or excessive induction time to achieve release.

Brief Summary Text (17):

Yet another significant object is to provide a process for making a <u>fertilizer</u> having one or more of the objects described above, or other objects that will be apparent, in a safe and cost efficient manner.

Brief Summary Text (18):

In a preferred embodiment of this invention, it has been found that one or more of the above-identified objects, and others, can be accomplished by utilizing an uncured raw linseed oil or other uncured suitable oil coating between the nutrient core and an encapsulating coating of <u>fertilizer</u> nutrient granules such as dicyclopentadiene-linseed oil. The raw linseed oil may be mixed with a binding agent such as a fine clay, diatomaceous earth or similar material before application to the nutrient core. If desired, the processing time of the manufacture of the <u>fertilizer</u> composition may be decreased by also including a drying agent such as a manganese/cobalt drier with the oil and clay mixture.

Brief Summary Text (19):

Use of clay with the linseed oil has been found to improve the process for producing coated granular fertilizer products. For example, without the clay, a large amount of fines, i.e., small, broken off bits of the coating, are produced by the tumbling action encountered by the particles during processing. Also, raw linseed oil alone tends to penetrate the dicyclopentadiene-linseed oil encapsulating coating, bleeding or migrating through the outer, encapsulating polymeric layer. The presence of the oil on the outside of this layer creates processing problems.

Brief Summary Text (20):

Attempting to solidify the linseed oil precoat by curing of the precoat to create a separate cured layer, as suggested by the Hansen '518 patent, prevents undue diffusion or migration of the linseed oil into the outer encapsulating layer. Indeed, curing the precoat appears to interfere with the physical interaction of the raw linseed oil precoat and the encapsulating coat. It has now been discovered that the presence of a raw linseed oil precoat, without being cured, contributes a significant improvement to coverage of granular nutrient surface defects by the subsequent, encapsulating layer and to the release characteristics of the fertilizer composition.

Brief Summary Text (21):

The current invention takes advantage of the previously unknown interaction between the unsolidified, free moving linseed oil, which may be mixed with a binding agent such as clay, diatomaceous earth and the like, and the encapsulating coat. The interaction provides release characteristics for the finished <u>fertilizer</u> product that otherwise would only be obtainable at even greater coating weights, particularly for substrates containing holes or other surface discontinuity or imperfections. Though not completely understood, it is currently believed that the interaction of the linseed oil and the polymeric layer more efficiently fills and plugs the surface holes, cracks and crevices of the core granule. This same interaction decreases the induction time.

Detailed Description Text (2):

The invention contemplates both a new fertilizer product and a new process for making such a product. Generally, the fertilizer particles are made of a nutrient core, a precoat and an outer or encapsulating coat. The core may be standard NPK or fertilizer granules as is well known in the art and which are commercially available from Norsk Hydro, Kemira and other companies. Alternatively, cores of other common nutrients (for example, urea) also can be used. In addition, the core may include one or more secondary nutrients such as calcium, magnesium and sulfur or micronutrients such as iron, copper, zinc, manganese, boron and molybdenum.

Detailed Description Text (3):

While the invention will work with relatively spherical granules having relatively smooth surfaces, use of the invention makes its greatest impact when controlled release <u>fertilizers</u> are made from irregularly shaped granules or those with surfaces containing holes, imperfections, cracks and crevices. An example of the relatively smooth nutrient core would be the Norsk NPK complex <u>fertilizers</u>. Nutrient granules with relatively greater number of discontinuities surface imperfections tend to be less expensive, are commercially available, and generally have sphericity of 50% or less. Hence, in a preferred embodiment of this invention, relatively inexpensive, low sphericity material may be used for the core nutrient.

Detailed Description Text (10):

The desired beneficial release characteristics of the fertilizer compositions of the present invention may be achieved by not exposing the precoat materials to temperatures too high to cure the oil after it is applied on the nutrient, but before the encapsulating layer is applied. In the preferred embodiment, in which the curing temperature for the linseed oil is similar to that for the polymeric encapsulating layer, the precoat layer may be simultaneously cured with the encapsulating material, in situ, after application of the encapsulating layer to the nutrient granule. Alternatively, it may be possible to cure the precoat after the encapsulating layer is applied, but before the encapsulating layer is cured.

Detailed Description Text (15):

For certain controlled release products, a maximum incremental release in the DDR-40 of 32 days, as shown for the <u>fertilizer</u> composition without a precoat, is undesirably long. For such products, 13% or less of imperfectly coated particles which release in the first 3 days, as reflected in the DDR-40 3 day percent release results, is acceptable.

Detailed Description Text (16):

In Table 1, the DDR-40 results of the pilot plant fertilizer composition No. 1, without a precoat, can be compared to pilot plant composition No. 2 which contains the precoat. Note the improvement in the DDR-40--3 days reported release from 16% to 10% with the linseed oil precoat. Also, the composition with the precoat showed a peak release at 14 days as compared to 32 days without the precoat. A similar improvement is shown for the production plant DDR-40 comparisons for compositions without a precoat, as compared to compositions having a linseed oil-clay precoat.

Detailed Description Text (20):

The incremental ART-80 release rates for a standard fertilizer in which no precoat was used is shown in FIGS. 1-3. Cumulative two hour release rates are the sum of the incremental rate at hour one plus the incremental rate at hour two. A high cumulative two hour release rate normally indicates too many imperfectly coated particles. The incremental release rate for the 6 PPH case bearing a relatively thin layer of OSMOCOTE.RTM. material is shown in FIG. 1. This fertilizer composition has been found to have a 17% cumulative release at two hours, an unacceptably high result. This suggests that the encapsulating coat did not sufficiently cover the surface imperfections on many of the granules. The 8 PPH case, shown in FIG. 2, also has cumulative release above the 4% specification at two hours. Finally, at 10 PPH, shown in FIG. 3, the product is below 4% at two hours for imperfectly coated particles.

CLAIMS:

- 1. A controlled release fertilizer comprising:
- a granular core of nutrient material;
- a preliminary coating of an organic oil applied on the granular core; and,
- a polymeric encapsulating coat applied over the preliminary coating on the granular core while the organic oil is uncured;

the preliminary coating being cured after application of the polymeric encapsulating coat.

- 2. The controlled release <u>fertilizer</u> of claim 1 wherein the organic oil is mixed with a binding agent selected from the group consisted of clays, talcs, diatomaceous earths, absorbent silicas, and mixtures thereof.
- 3. The controlled release $\underline{\text{fertilizer}}$ of claim 1 wherein the granular core has sphericity of 50% or less.
- 4. The controlled release <u>fertilizer</u> of claim 3 wherein the organic oil is raw linseed oil.
- 5. The controlled release $\underline{\text{fertilizer}}$ of claim 1 wherein the binding agent is a fine clay.
- 6. The controlled release <u>fertilizer</u> of claim 2 wherein the preliminary coating further includes a drier.
- 7. The controlled release <u>fertilizer</u> of claim 6 wherein the drier is selected from the group consisting of cobalt manganese, manganese, cobalt calcium, zirconium cobalt, and mixtures thereof.
- 8. The controlled release fertilizer of claim 1 wherein the core is selected from

the group consisting of NPK granules, $\underline{\text{fertilizer}}$ granules, urea granules, and mixtures thereof.

- 9. The controlled release <u>fertilizer</u> of claim 8 in which the core further contains one or more secondary nutrients and <u>micronutrients</u> selected from the group consisting of calcium, sulfur, magnesium, iron, copper, zinc, manganese, boron and molybdenum.
- 10. The controlled release <u>fertilizer</u> of claim 1 wherein the core has sphericity of 50% or less and the controlled release <u>fertilizer</u> exhibits a 4% or less cumulative release at two hours in the ART-80 laboratory test when the encapsulating coat is at 8 parts per hundred of the core weight or less.
- 11. The controlled release <u>fertilizer</u> of claim 10 wherein the maximum incremental ART-80 laboratory test release is at 15 hours or less.
- 12. A controlled release <u>fertilizer</u> comprising:
- a granular core of nutrient material;
- a preliminary coating on the granular core of a mixture of (a) an oil selected from the group consisting of linseed oil, soybean oil, tung oil, dicyclopentadiene modified drying oils, and mixtures thereof and (b) a binding agent selected from the group consisting of fine clays, talcs, diatomaceous earths, absorbent silicas, and mixtures thereof; and,
- a polymeric encapsulating layer applied over the coating;

the oil in the preliminary coating mixture being uncured; and

the preliminary coating being cured after application of the polymeric encapsulating layer to the coated granular core.

- 13. The controlled release <u>fertilizer</u> of claim 12 wherein the polymeric encapsulating coat is a copolymer of dicyclopentadiene and a composition selected from the group consisting of linseed oil, an alkyd resin based on a soybean oil, and mixtures thereof.
- 14. The controlled release <u>fertilizer</u> of claim 12 wherein the core has sphericity of 50% or less and the controlled release <u>fertilizer</u> exhibits a 4% or less cumulative release at two hours in the ART-80 laboratory test when the encapsulating coat is at 8 parts per hundred of the core weight or less.
- 15. A controlled release $\underline{\text{fertilizer}}$ comprising a nutrient core, a coating of organic oil and an encapsulating polymeric layer produced by a process comprising the steps of:
- (a) applying the organic oil on the nutrient core while the organic oil is uncured;
- (b) applying the polymeric layer over the organic oil before the organic oil is cured; and,
- (c) simultaneously curing the organic oil and the polymeric layer.
- 16. The controlled release $\underline{\text{fertilizer}}$ of claim 15 wherein the organic oil is applied at about 140.degree. F. or $\overline{\text{less}}$.
- 17. The controlled release <u>fertilizer</u> of claim 16 wherein the simultaneous curing occurs at about 140.degree. F. or above.
- 18. The controlled release <u>fertilizer</u> of claim 15 wherein the organic oil is mixed with a binding agent selected from the group consisting of fine clay, talc, diatomaceous earth, absorbent silicas, and mixtures thereof.
- 19. The controlled release <u>fertilizer</u> of claim 18 wherein the organic oil is further

- mixed with a drier selected from the group consisting of cobalt manganese, manganese, cobalt calcium, zirconium cobalt, and mixtures thereof.
- 20. The controlled release <u>fertilizer</u> of claim 18 wherein the oil is raw linseed oil.
- 21. The controlled release $\underline{\text{fertilizer}}$ of claim 20 wherein the binding agent is a fine clay.
- 22. The controlled release <u>fertilizer</u> of claim 21 wherein the polymeric encapsulating coat chosen from the group consisting of oleoresinous drying oils, polyesters, polyamides, polyurethanes, thermoplastic resins, dicyclopentadiene modified linseed oil, dicyclopentadiene modified allyd resins, hydrocarbon thermoplastic resins, and mixtures thereof.
- 23. The controlled release <u>fertilizer</u> of claim 22 in which the core has sphericity of 50% or less and the controlled release <u>fertilizer</u> exhibits a 4% or less cumulative release at two hours in the ART-80 laboratory test when the encapsulating coat is at 8 parts per hundred of the core weight or less.
- 24. A controlled release <u>fertilizer</u> containing a nutrient core, a precoat of linseed oil, a binding agent and a drier, and a polymeric encapsulating layer including dicyclopentadiene, the <u>fertilizer</u> having been produced by the steps of:
- (a) coating the core with the precoat at about 140.degree. F. or less while the linseed oil is uncured;
- (b) encapsulating the precoat with the polymeric layer; and,
- (c) simultaneously curing the precoat and the encapsulating polymeric layer at about 140.degree. F. or above.
- 25. The controlled release <u>fertilizer</u> of claim 24 wherein the core has sphericity of 50% or less and the controlled release <u>fertilizer</u> exhibits a 4% or less cumulative release at two hours in the ART-80 laboratory test when the encapsulating coat is at 8 parts per hundred of the core weight or less.
- 26. A process of making a controlled release <u>fertilizer</u> utilizing a nutrient core, the process consisting of the steps of:
- (a) precoating the nutrient core with oil, the oil selected from the group consisting of linseed oil, soybean oil, tung oil, dicyclopentadiene modified drying oils, and mixtures thereof;
- (b) applying an encapsulating layer of polymeric material over the precoated nutrient core, the polymeric layer being applied before the precoat is cured and while the oil is uncured; and,
- (c) curing the precoat after application of the encapsulating layer.
- 33. The process of claim 26 wherein the core has sphericity of 50% or less and the controlled release <u>fertilizer</u> exhibits a 4% or less cumulative release at two hours in the ART-80 laboratory test when the encapsulating coat is at 8 parts per hundred of the core weight or less.
- 34. A process for making a controlled release <u>fertilizer</u> utilizing a core of nutrient having surface discontinuities, the process consisting of the steps of:
- (a) precoating the nutrient core with a mixture of raw linseed oil, a binding agent and a drier, the precoating applied in a proportion of about 0.5-3.0 parts per hundred of the weight of the core and being exposed to temperatures in the range of 140.degree. F. or less while the linseed oil is uncured;
- (b) applying an encapsulating layer of polymeric material before the precoat is cured, the encapsulating layer being applied in a proportion of about five to ten

parts per hundred of the weight of the core; and,

- (c) curing the precoat and the encapsulating layer at 140.degree. F. or more.
- 35. The process of claim 34 wherein the core has sphericity of 50% or less and the controlled release <u>fertilizer</u> exhibits a 4% or less cumulative release at two hours in the ART-80 laboratory test when the encapsulating coat is at 8 parts per hundred of the core weight or less.

WEST

Generate Collection Print

L12: Entry 3 of 10

File: USPT

Dec 15, 1998

DOCUMENT-IDENTIFIER: US 5849060 A

TITLE: Controlled release fertilizer and preparations thereof

Assignee Name (1):

Zhengzhou Centre of Popularization & Research on Zhengzhou Luxuriance Phosphate & Compound Fertilizer

Assignee Group (1):

Zhengzhou Centre of Popularization & Research on Zhengzhou Luxuriance Phosphate & Compound Fertilizer CN 03

Abstract Text (1):

The present invention relates to a controlled release <u>fertilizer</u> having water-soluble <u>fertilizers</u> as nucleus and limited-soluble plant nutrient compounds as coating layers, characterized in that more than two coating layers of limited-soluble compounds are formed around the nucleus, and that the release rates of the <u>fertilizer</u> can be controlled by regulating the solubilities of the limited-soluble compounds. The invention also relates to a process for preparing the controlled release fertilizer.

Brief Summary Text (2):

The present invention relates to a controlled release <u>fertilizer</u> and the methods for preparing the same.

Brief Summary Text (4):

A controlled release <u>fertilizer</u> is a kind of compound <u>fertilizer</u> which can control the release of plant nutrients therefrom.

Brief Summary Text (5):

Nowadays, people have paid more attention to development of various kinds of compound fertilizer so that the plant nutrients contained can be slowly released within a required time, and that the release rate can be consistent with rates of plants absorption during their growing stages. Since 1960s, various coated fertilizers have been developed in U.S.A, Japan, England, France etc., of which sulfur coated fertilizer and resin coated fertilizer are two of the main types.

Brief Summary Text (6):

Sulfur coated fertilizer was first developed by U.S.A in 1961, and came into commercial production in 1978. In 1990, the output of the fertilizer in the world had amounted to 115,000t which was below 0.2 percent of the amount of the total urea output in the world. In 1975, the sulfur coated compound fertilizer was developed by Japan; and in 1981, sulfur coated potassium chloride and sulfur coated potassium sulfate fertilizers were developed by O. M. SCOTT, U.S.A. Processes for coating sulfur are very complicated in that melted sulfur is first coated on the surfaces of urea granules, and then the sulfur coated surfaces are sealed with melted paraffin wax. The weakness of this kind of fertilizer is that the nucleus of the fertilizer is released out very quickly and that the coated wax is easy to be decomposed by microorganisms in the soil, as a result of which the controlled release effect is disadvantageously affected.

Brief Summary Text (7):

The coating layer of a resin coated slow release fertilizer represented by the

OSMOCOTE.RTM. U.S.A is essentially composed of a copolymer of cyclopentadiene dimer and glyceride, the amount of the coating layer is at a level of about 10-15% wt of the total weight of <u>fertilizer</u>. The resin coated slow release <u>fertilizer</u> has excellent controlled release properties. The contained plant nutrients can be controlled to release out 80% of the nutrients in 120-360 days.

Brief Summary Text (8):

The common disadvantage of the above-said two types of coated <u>fertilizers</u> is that they are expensive. For example, according to U.S.A market price in 1994, the price of SCU was 1.7 times as much as that of the urea, and the resin coated <u>fertilizer</u> was 8.2 times as much as that of the urea.

Brief Summary Text (9):

On the other hand, a coated compound <u>fertilizer</u> was disclosed in China Patent CN 851010089 which comprises the granular urea or ammonium nitrate as nucleus coated with a <u>fertilizer</u> selected from FMP (fused magnesium phosphate), precipitated calcium phosphate and bone powder, with the use of nitrogen phosphorus slurry as binders.

Brief Summary Text (10):

This kind of fertilizer whose cost is greatly decreased with the use of fertilizer coated with fertilizer, was honored as "the cheapest slow release fertilizer came from China" by an international magazine ("Nitrogen", No. 191, pages 35-41, May-Jun., 1991). The price of the fertilizer in China is only 75 percent of that of urea. However, under the electronic microscope, the coating layer of the fertilizer observed shows a slice structure with many large and small holes and cracks on surfaces of the fertilizer, which can not prevent water from penetrating the coating layer effectively. When soaked in water, the coated urea and ammonium nitrate in the nucleus of the fertilizer will be dissolved out completely in 1.5-2.0 hours so that a good controlled release effect can not be achieved.

Brief Summary Text (11):

As an improvement a slow release coated <u>fertilizer</u> is disclosed in Chinese Patent CN89102295, which comprises using vegetable oils, animal oil or their greases or their modified substances as a retarder in the coating layer so that the release time of nutrients in the nucleus can be prolonged to 15-30 days, by this way, the release of nutrients in the nucleus can be retarded. But there are some shortcomings:

Brief Summary Text (12):

a. The release of water soluble potassic fertilizer can not be retarded.

Brief Summary Text (13):

b. Adding so many retarders which are not plant nutrients into <u>fertilizer</u> will affect the total nutrient content in the fertilizer,

Brief Summary Text (15):

It is therefore an object of the present invention to provide a type of compound fertilizer composed of compounds containing nitrogen, phosphorus, potassium, magnesium and trace elements with various release rates. By regulating the structure and the composition of the coating layers, solubilities of the limited-soluble compounds can be changed, and release rate of nutrients can be controlled so as to satisfy with the desire of plants in different growing stages.

Brief Summary Text (16):

It is another object of the invention to provide a method for preparing said controlled release fertilizer.

Brief Summary Text (18):

The present invention provides a controlled release <u>fertilizer</u> with water-soluble <u>fertilizer(s)</u> as nucleus and limited-soluble plant nutrients as coating layers, characterized in that coating layers of limited-soluble compounds are formed around the nucleus, and that release rates of the nutrients can be controlled by regulating the solubilities of the limited-soluble compounds. The present invention also provides methods for preparing the abovementioned <u>fertilizers</u>, which comprise the

following steps mainly:

Brief Summary Text (19):

a) selecting one or more fertilizer(s) as nucleus;

Drawing Description Text (4):

FIG. 3 shows the nitrogen releasing curve of the <u>fertilizers</u> produced according to examples 1 and 2.

Detailed Description Text (2):

The coating layer of the controlled release fertilizer according to the present invention can be formed by using a mixture of sulfuric acid or phosphoric acid at certain concentrations with monoammonium phosphate (MAP); diammonium phosphate (DAP) or ammonium bicarbonate as binders to coat with compounds with various solubilities on the surface of water-soluble granular fertilizers layer by layer; or by using reactive binders to coat with compounds containing nitrogen, phosphorus, potassium, magnesium and trace element on the surface of water-soluble fertilizers, during which period limited-soluble compounds are formed in the form of several coating layers. Water-proof substances such as silicone or calcium carbonate stearate can be added into the coating layers to enhance the hydrophobicity. And outside the coating layers reactive magnesium oxide or phosphate rock powder can be added to form fine and close shells. The present invention also provides use of the controlled release fertilizers in lawns (especially golf course) and ornamental plant.

Detailed Description Text (9):

47.5 kg of magnesium oxide (85% in purity, industrial grade) was added to 355 kg of phosphoric acid (20% of P.sub.2 O.sub.5, industrial grade). The reaction temperature was maintained at 85.degree. C. for 0.25 hour with continuously stirring to form a suspension of MgHPO.sub.4.3H.sub.2 O and MgO with a pH value of 2. Continuously adding with ammonia the pH value of the suspension was increased to a level from 5 to 6, resulting in a suspension containing MgNH.sub.4 PO.sub.4. H.sub.2 O and MgO, after filtering, obtaining a coating material of a controlled release fertilizer which contains nitrogen 10.2 wt %, P.sub.2 O.sub.5 45.9 wt %, MgO 26.2 wt % ("Phosphate and Compound fertilizer", No.4, pp19-20, 1989). Compounds 10-12 in Table 1 are commercially available products and compound 13 is formed by the absorption of carbon dioxide in air on magnesium hydroxide. The plant nutrients, such as nitrogen, phosphorus, potassium, magnesium, zinc and manganese contained in the above compounds with limited solubilities in water can be slowly released in water. The coating layers composed of the above compounds are in an amount of 35 to 68 percent on the basis of the total weight of the fertilizer.

Detailed Description Text (10):

2. Selection of the binders. The binders used comprise non-reactive binders and reactive binders. The non-reactive binders can be a mixture of sulfuric acid or phosphoric acid at certain concentrations with monoammonium phosphate, diammonium phosphate or ammonium bicarbonate, which mixture is bound with compounds having various solubilities and coated on the surface of water-soluble granular fertilizer layer by layer. On the other hand, the binders can be reactive binders which can react with single fertilizer such as urea, monoammonium phosphate, diammonium phosphate, ammonium bicarbonate, ammonium sulfate, potassium chloride or potassium sulfate to form limited-soluble compounds . Two kinds of reactive binders can be selected, one kind is organic binders such as formaldehyde, starch and carboxy methyl cellulose, which can condense with urea to form limited-soluble compounds containing nitrogen. Urea has a property of condensation-addition with various organic compounds (UREA, ITS PROPERTIES AND MANUFACTURE). Now an example is taken for illustrating the condensation reaction of urea with formaldehyde. 0.04 kg hexamethyleneamine was added to 3kg formaldehyde solution (37 wt %), after completely dissolved, 0.8 kg urea was added with continuously stirring and heating until said urea was completely dissolved. The mixture was heated to a constant temperature of 60.degree. C. for 15 min., then the temperature was maintained at 92.degree. C. for 70 min., so that urea-formaldehyde condensation was taken place, 0.4 kg aqueous urea solution (50 wt %) was added in 30 min. to keep on the reaction. After 20 min., the reaction was stopped. The reaction solution was cooled to 60.degree. C. and then a 10% aqueous sodium hydroxide solution was added to neutralize the urea-formaldehyde resin solution till pH=7, and then the reaction

mixture was cooled to room temperature to obtain a semi-transparent slurry, which was then mixed with water in a ratio of 1:5 to get urea-formaldehyde resin binders. The other kind of binders is inorganic binders such as phosphoric acid or sulfuric acid used alone or mixed with monoammonium phosphate, diammonium phosphate, ammonium bicarbonate or ammonium sulfate, said binders can react with magnesium oxide, calcium oxide, zinc sulfate, manganese sulfate, potassium sulfate or potassium chloride to obtain compounds 1-9 in Table 1. The concentrations and formulations of the selected binders are shown in Table 2. The amount of the binders is 5-10% on the basis of the total weight of fertilizer.

Detailed Description Text (11):

3. The solubilities of limited-soluble compounds 1-10 in Table 1 can be adjusted by adding inorganic additives such as ammonium sulfate, ammonium chloride, ammonium nitrate and magnesium sulfate into the coating layer and the release rates of nutrients in the <u>fertilizer</u> can be controlled. The amount of regulator is 0-5% on the basis of the total weight of <u>fertilizer</u>. For example, the solubility of MgNH.sub.4 PO.sub.4.6H.sub.2 O in Table 1 can be adjusted by adding ammonium sulfate as shown in Table 3. When no ammonium sulfate is added, 80% nitrogen in the <u>fertilizer</u> can be released out within 140 days. If adding 5% ammonium sulfate, the release period is only 70 days.

Detailed Description Text (12):

The solubilities of the limited-soluble compounds can also be changed by adjusting the pH values of the salts contained in the coating layers so as to control the release rates of the <u>fertilizers</u>. For example, the solubilities of FMP listed in Table 1 will change along with the pH values of the solutes, as shown in Table 4.

Detailed Description Text (14):

4. Treatment of the toppest layer of the coating layers can increase the density of the coating layers and decrease the hydrophilicity of the coating layers. Therefore, powder is applied on the surface of the coating layer. The powder used comprises fine magnesium oxide powder(<0.075 mm), which absorbs carbon dioxide from air to form compound 13 in Table 1 with smooth and fine shells. Alternatively, a calcium carbonate stearate powder (a water proof and insulated powder material whose preparing method can be found in Chinese Patent CN 91105131.7) is applied or a organic silicone is coated on to form a hydrophobic layer. The amount of the powder and hydrophobic layer applied is 2-5% by weight of the total fertilizer.

Detailed Description Text (15):

As stated above, the controlled release fertilizer according to the present invention can be manufactured by using granular water-soluble fertilizer such as urea, ammonium nitrate monoammonium phosphate, diammonium phosphate, potassium chloride, potassium sulfate and other compound fertilizer as nucleus, and coating on the nucleus with compounds containing nitrogen, phosphorus, potassium, magnesium and trace elements and having different limited solubilities layer by layer; or coating on the nucleus with limited soluble compounds which are formed by the reaction between a reactive binder and a single fertilizer during coating process. The number of coating layers are not less than 2, and preferable 3-5 so that the coating layers have a desirable thickness and different release rate. The solubility of limited-soluble compound can be changed by regulating the composition and pH value of coating layers, by which nutrient-releasing rate can be controlled to a release period of 60-240 days (the time required to release 80% nutrients). A controlled release fertilizer containing 30-50 wt % nitrogen, phosphorus pentoxide, potassium oxide and magnesium oxide can be manufactured optionally. Analysis with a scanning electroscope found that the cross section of coating layers was composed of dense amorphous compounds.

Detailed Description Text (16):

The controlled release <u>fertilizer</u> is essentially composed of nucleus and coating layers, the amount of nucleus is 20-50% by weight of the total mass, while the amount of coating layer is 50-80% by weight of the total mass. And the coating layer is comprised of a limited-soluble compound, an adjustment agent, a powder and a binder, which are 35-70%, 0-5%, 2-5% and 5-10% by weight of the total <u>fertilizer</u> respectively most of which are plant nutrients.

Detailed Description Text (20):

35 parts of granular urea was put into a special CCF granulator (China patent number: ZL892 14248.0), and preheated to 60.degree. C. by blowing hot air (70.degree. C.) heated by an electric heater. Then 1.3 parts formaldehyde (37 wt. %) solution containing hexamethyleneamine was sprayed into the granulator by an atomizer, after the granular urea was wetted, 3.6 parts powder urea was continuously added during the spraying of formaldehyde to form the first coating layer of urea-formaldehyde which was 5% by weight of the fertilizer; 17 parts reactive magnesium oxide and 7 parts monoammonium phosphate powder were added to the granules having the first coating layer by means of IC binder (which was prepared, as shown in Table 2, by adding 5 parts 40% phosphoric acid (based on P.sub.2 O.sub.5) to 2.6 parts ammonium bicarbonate), to provide the second coating layer containing ammonium magnesium phosphate, which was 26.6% by weight of the fertilizer; and then the third coating layer containing magnesium potassium phosphate, which was 26% by weight of the <u>fertilizer</u>, was formed by using a blend solution of 20% sulfuric acid and 40% phosphoric acid (based on P.sub.2 O.sub.5) in a ratio of 7:3 as binder, and continuously adding 5 parts monoammonium phosphate and 21 parts potassium chloride powder to the granules having the second coating layer. Finally, 5 parts calcium carbonate stearate was added to provide a hydrophobic layer which was 5% by weight of the total fertilizer. The final product was dried at 70.degree. C. until the water content was less than 2%, and packed up after cooling to obtain a controlled release fertilizer with four coating layers. Measurment with a scanning electroscope found that the average diameter of nucleus was 2,300 .mu.m, and the average total thickness of the four coating layers was 740 .mu.m, and the average diameter of the fertilizer granule was 3780 .mu.m. The composition of the fertilizer was N19%, P.sub.2 O.sub.5 10%, K.sub.2 O 12%, MgO 10%. Analysis by x-ray diffraction proved that the coating layer contained MgO, NH.sub.4 MgPO.sub.4.6H.sub.2 O, Mg(OH).sub.2, KMgPO.sub.4.6H.sub.2 O, KCaPO4.H.sub.2 O (see FIG. 2).

<u>Detailed Description Text</u> (22):

10 parts sulfuric acid (10% in concentration) was placed into an acid-proof reaction tank, to which 5 parts monoammonium phosphate powder was added gradually under continuously stirring at ambient temperature, standing for half an hour a binder solution (ID in table 2) was obtained; 33 parts of granular ammonium nitrate was placed into a granulator, into which binder ID was sprayed and 25 parts NH.sub.4 MgPO.sub.4.6H.sub.2 O were added to provide the first coating layer which was 27% by weight of the total fertilizer. To a solution containing 10 parts 5% carboxymethyl cellulose, 4 parts urea powder was added, the mixture was allowed to react for 3 hours to provide binder OB in Table 2. The second coating layer was formed by coating 16 parts synthesized KMgPO.sub.4.H.sub.2 O onto the granules having the first coating layer by means of binder OB, which was 17.7% by weight of the total fertilizer. The third coating layer was formed by coating with 16 parts FMP by means of 30 wt & sulfuric acid as binder, which was 18% by weight of the total fertilizer. Finally, a fine and close shell layer was formed by powdering on the surface of the fertilizer with 4 parts magnesium oxide powder. The final product was dried at 70 C until the water content was less than 2%, and then packed up after cooling to obtain a fertilizer containing N 13%, P.sub.2 O.sub.5 18%, K.sub.2 O 5% and MgO 3%.

<u>Detailed Description Text</u> (23):

The release rate of the fertilizer in soil was determined as follow:

Detailed Description Text (25):

The <u>fertilizer</u> sample of example 2(3 g) was incorporated uniformly into the simulated soil. The medium was then surface irrigated with distilled water until the medium was nearly saturated, but with little or no leaching from the pot. The whole installation was sealed and held at a constant temperature of 20.degree. C. After a period of time, the saturated solution was extracted and analyzed for the N content, and resaturated with distilled water. The various release rates at different time can be measured in this way. The release rates curves was shown in FIG. 3.

Detailed Description Text (26):

Effect of the Fertilizer of the Invention

<u>Detailed Description Text</u> (27):

The main advantages of the fertilizer according to the present invention are as

Note: (1)

follows:

Detailed Description Text (28):

The Fertifizer provides 60, 90, 120 days longevity for nitrogen releasing, high utilization ratio of fertilizer, less frequent application and reduced labor costs;

Detailed Description Text (29):

it provides fully plant nutrients, need not blend with other <u>fertilizers</u>, it is easy for application, it feeds N, P, K and trace elements at one-shot according to the plant requirements;

Detailed Description Text (31):

high safety for use and no pollutant, no plastic or wax contained in said <u>fertilizer</u> and all the components are plant nutrients;

Detailed Description Text (33):

The <u>fertilizer</u> of the invention can be widely used in the fairways, roughs, parks, race courses, lawn tennis courts, sport grounds, turf farms and lawns.

<u>Detailed Description Paragraph Table</u> (6):

TABLE 1

The present <u>fertilizer</u> Trial Results vs Scotts on Small Plots Trial Set June 1, 1995 The present <u>Lbs.N</u> July 1 Aug 1 Sept 1 Oct 1 Nov 1 Longevity <u>fertilizer</u> per 1000 Ft.sup.2 June 1 30days 61days 92days 122days 153days days

1.0# Set 9 10 10 7 5 122 22-2-10 1.0# Set 10 10 10 8 6 122 18-6-12 1.5# Set 10 10 10 7 5 122 22-2-10 1.5# Set 10 10 10 9 7 153 18-6-12 2.0# Set 9 10 10 8 8 153 22-2-10 2.0# Set 10 10 10 9 8 153 Scotts PolyS 1.0# Set 10 10 7 6 4 92 21-3-20 100% PolyS 1.0# Set 10 10 7 7 5 122 26-4-12 100% PolyS 1.0# Set 9 10 7 5 4 92 21-0-20 100% PolyS 1.5# Set 10 10 7 5 5 92 21-3-20 26-4-12 1.5# Set 10 9 8 7 6 122 21-0-20 1.5# Set 10 10 7 5 5 92 21-3-20 2.0# Set 10 9 9 7 5 122 26-4-12 2.0# Set 10 10 7 5 122 21-0-20 2.0# Set 10 10 9 7 5 122

Overall evaluation 1-10 growth color density (2) Blue grass with come bent and rye (3) Healthy turf (4) Good rainfall (5) Reapplication would occur at 7 (6) "The present fertilizer" in all tables was made according to example of the invention. The type number "ab-c" means that the contents of nitrogen, phosphorus and potassium are a%, b% and c% respectively. For example "186-12" means the contents of nitrogen, phosphorus and potassium are 18%, 6% and 12% respectively. And the type number has the same meanin in comparable examples.

Detailed Description Paragraph Table (7):

TABLE 2

Tabulated From Field sheet-by D. Deirson-Rate Trial of the present <u>fertilizer</u> Trail set 11/7/95 The present Lbs of N 12/8/95 15/9/95 6/10/95 4/11/95 <u>fertilizer</u> Lbs/1000 ft (32 days) (66 days) (87 days) (116 days) Snow Comment

Detailed Description Paragraph Table (8):

TABLE 3 - Trials of the present <u>fertilizer</u> compared with other controlled release <u>fertilizer</u> in USA Trail Conditions Trials on Grass in USA Lbs of N Treated <u>Evaluation</u> Criterion Location of Grass (Lbs/1000 area Irri- Uniform Stress No. Trial

Place type Date Product ft.sup.2) (ft.sup.2) gated Color Density growth Tolerance Comment 1 Bay area Alameda Blue 1/6/95 The present <u>fertilizer</u> 1.5 1000 .smallcir cle. 10 9 9 9 1. The present <u>fertilizer</u> California Golf Rye (31days) (20-2-10) 1.5 1000 .smallcircle. 10 8 9 9 lasted 120 days effective 119.degree.30'W Club IBDU (21-3-16) 2. IBDU lasted 90 days 37.degree.30N 3/8/95 The present <u>fertilizer</u> 1.5 1000 .smallcircle. 10 10 9 8 effective (93 days) (20-2-10) 1.5 1000 .smallcircle. 7 9 9 5 3. No rainfall IBDU (21-3-16) 5/9/95 The present fertilizer 1.5 1000 9 8 8 8 (120 day) (20-2-10) 1.5 1000 5 6 6 4 IBDU (21-3-16) 2 Tuf-Turf Land- Blue 6/7/95 The present fertilizer 3/4 1000 x 10 9 9 9 1. The present fertilizer Kansas scape Rye (30 days) (20-2-10) Scotts 3/4 1000 x 10 9 9 9 lasted 120 days effective 94.degree.38'W Co. Poly-S (21-2-21) 2. Scotts longevity lasted 39.degree.07N 7/8/95 The present <u>fertilizer</u> 3/4 1000 x 9 9 9 60 days (72 days) (20-2-10) Scotts 3/4 1000 x 7 7 8 7 3. Heavy rain 15 in. in month Poly-S (21-2-21) reduced longevity of both 10/9/95 The present fertilizer 3/4 1000 8 7 7 products. (120 day) (20-2-10) Scotts 3/4 1000 5 5 5 Poly-S (21-2-21) 3 Nebraska Dokoto Blue 14/8/95 The present fertilizer 1.5 1000 .smallcir cle. 8 7 7 7 100.degree.00'W City Rye (20-2-10) 1.5 1000 .smallcircle. 8 8 7 7 41.degree.31'N 7WIR (16-2-13) The present fertilizer 3/4 1000 .smallcircle. 8 8 8 8 (20-2-10) 3/4 1000 .smallcircle. 8 8 8 7WIR (16-2-13) The present fertilizer 1 1500 .smallcircle. 7 7 7 7 (20-2-10) 7WIR (16-2-13) 1 500 .smallcircle. 6 6 6 6 The present fertilizer 3/4 2000 x 5 6 5 5 (20-2-10) 3/4 2000 x 5 6 6 5 7WIR (16-2-13) 4 Texas Waco Burmu- 7/8/95 The present fertilizer 1.8 2000 .smallcircle. 10 10 10 Product lasted in hot 94.degree.54'W da (34 days) (20-2-10) condition 138 days before 29.degree.24'N 4/10/95 The present fertilizer 1.8 2000 .smallcircle. 10 10 10 cooling took place. (95 days) (20-2-10) 16/11/95 The present fertilizer 1.8 2000 .smallcircle. 10 10 10 (138 day) (20-2-10) 10/8/95 The present fertilizer 1.5 6600 x 10 10 10 10 1. LUXACOTE lasted (30 days) (20-2-10) Scotts 1.4 10000 x 10 10 10 10 120 days and still remained Poly-S (25-3-10) 3.0 5600 x 9 10 10 10 green. ONCE(34-4-7) Ollio urban Blue 14/9/95 The present fertilizer 1.5 6600 x 10 10 10 10 2. Cool weather slowed IIlinois Golf grass (65 days) (20-2-10) Scotts 1.4 10000 x 10 10 10 9 growth (grass). Club Poly-S (25-3-10) 3.0 5600 x 10 10 10 10 ONCE(34-4-7) 5 89.degree.00'W School Fescue 15/10/95 The present fertilizer 1.5 6600 x 10 10 10 10 3. Scott 100% Poly-S did 40.degree.00'N football (96 days) (20-2-10) Scotts 1.4 10000 x 10 10 9 9 not last 120 days as fields Poly-S (25-3-10) expected. ONCE(34-4-7) 3.0 5600 x 10 10 10 10 12/11/95 The present $\underline{\text{fertilizer}}$ 1.5 6600 x 9 10 9 9 (20-2-10) Scotts 1.4 10000 x 7 8 7 7 (123 day) PoIy-S (25-3-10) 3.0 5600 x 10 10 9 9 ONCE(34-4-7) 6 Carmel Terra Ftcue 11/7/95 The present fertilizer 1/2 1000 1. Lasted 120 day. Indiana office (20-2-10) 1 1000 2. Rich Grant reported (trial by that LUXACOTE was Rich effective for 120 day. Grant) 86.degree.15 W Blue The present fertilizer 1/2 1000 3. Trial conducted in 40.degree.00'N Rye (18-6-12) 1 1000 landscape situation at back of terra office. 7 Research 24/7/95 The present fertilizer 2 5000 .smallcircle. 5 3 7 My understanding that LUXACOTE station (20-2-10) maintained good performance untill The present fertilizer 1 5000 .smallcircle. 10 10 10 fall cool season. (20-2-10) 50% Poly-S(24-4-16) 2 5000 .smallcircle. 7 3 3 50% Poly-S(24-4-16) 1 5000 .smallcircle. 3 3 7 8 Michigan Gay Bent 1/10/95 The present fertilizer 1 1000 9 9 9 9 1. Evaluated after 90 days, 86.degree.54'W Lord Blue (60 day) (20-2-10) Two cool weather in oct. stoped 41.degree.43'N Country compete grass growthlooked good Club Fairway until snow. 2. Provided equal results after 90 days with 100% Poly-S product 3. Would use again next year. No burning or darmage on bent fairway.

Other Reference Publication (1):

Zhang Baolin et al., "Luxuriance: A slow-acting compound fertilizer from China", Nitrogen No. 191 (May/Jun. 1991).

CLAIMS:

1. A controlled release granular <u>fertilizer</u> having water-soluble <u>fertilizers</u> as a nucleus and limited-soluble plant nutrient compounds as coating <u>layers</u>, wherein more than two coating layers of limited-soluble compounds are formed around the nucleus, by bonding the nucleus with the coating layers having various solubilties by means of an aqueous 6-30 wt % sulfuric acid or 30-60 wt % phosphoric acid (based on P.sub.2 O.sub.5) solution mixed with monoammonium phosphate, diammonium phosphate, or ammonium bicarbonate as a binder, and the release rates of the nutrient compounds are controlled by regulating the solubilities of the limited-soluble compounds.

- 2. A controlled release granular fertilizer having water-soluble fertilizers as a nucleus and limited-soluble plant nutrient compounds as coating layers, wherein more than two coating layers are formed around the nucleus by bonding the nucleus with the coating layers by means of an inorganic reactive binder comprising an aqueous 6-30 wt % sulfuric acid or 30-60 wt % phosphoric acid (based on P.sub.2 O.sub.5) solution alone or a mixture of an aqueous sulfuric acid or phosphoric acid solution mixed with monoammonium phosphate, diammonium phosphate, ammonium bicarbonate or ammonium sulphate, said limited-soluble plant nutrient compounds are formed by reacting compounds containing nitrogen, phosphorous, potassium, magnesium and trace elements selected from the group consisting of MgO, CaO, ZnO, MnSO.sub.4, KCL, and K.sub.2 SO.sub.4 with said inorganic reactive binder during coating; and the release rate of the nutrient compounds are controlled by regulating the solubilities of the limited-soluble compounds.
- 3. The controlled release <u>fertilizer</u> according to claim 1 or 2, wherein the solubilities of the limited soluble compounds are adjusted by adding at least one inorganic salt selected from the group consisting of ammonium sulphate, ammonium chloride, ammonium nitrate, and magnesium sulphate, and/or by regulating pH values of the coating layers.
- 4. The controlled release $\frac{\text{fertilizer}}{\text{fertilizer}}$ according to claim 1 or 2, wherein the surfaces of the controlled release $\frac{\text{fertilizer}}{\text{fertilizer}}$ are treated with silicone or calcium carbonate stearate to enhance the hydrophobicity of the coating layers, and magnesium oxide is used as a powder to smooth the surfaces.

Generate Collection Print

L12: Entry 4 of 10 File: USPT Oct 22, 1996

DOCUMENT-IDENTIFIER: US 5567221 A

TITLE: Compositions and methods for use in aquaculture

Abstract Text (1):

Controlled release <u>fertilizer</u> compositions which release <u>fertilizer</u> at a controlled rate over an extended period of time are employed in aquaculture treatment methods whereby nutrients are efficiently and effectively released into closed aquatic ecosystems such as ponds, lakes, watersheds and other aqueous environments over a period of time in order to enhance the growth of phytoplanktonic algae populations in the water and to thereby promote marine life populations.

Brief Summary Text (3):

This invention relates to compositions and methods for use in aquaculture. More particularly, it relates to methods employing controlled release <u>fertilizer</u> compositions to release nutrients into closed aquatic ecosystems such as ponds, lakes, watersheds and other aqueous environments in order to enhance the growth of phytoplanktonic algae populations in the water and to thereby promote marine life populations.

Brief Summary Text (5):

Fertilization has been recognized heretofore as being an important technique in management of closed aquatic ecosystems such as ponds, lakes, watersheds and the like, particularly for purposes of stimulating the growth of phytoplanktonic algae in the water. Such phytoplanktons serve as the basis of the food chain in such aquatic environments and are, therefore, necessary for increasing fish production in these closed aquatic ecosystems. In addition, proper fertilization techniques have been shown to serve several other useful functions including limiting the growth of troublesome aquatic weeds in the closed aquatic environment and improving water quality.

Brief Summary Text (6):

For most freshwater ponds and lakes, phosphorus is considered to be a key nutrient in fertilizers, but inclusion of nitrogen and other nutrients in the fertilizers has been recognized to be desirable. However, phosphorus is the limiting nutrient for development of phytoplanktonic algal blooms. Thus, most ponds require frequent addition of fertilizers rich in phosphorus to maintain good phytoplankton blooms throughout a production cycle. Frequent fertilization with phosphorus is needed because available phosphorus is quickly adsorbed by pond muds or taken up by phytoplankton. Once adsorbed onto pond muds, little phosphorus is released back into pond waters, because the oxidized water-mud interface acts as a barrier between mud and overlying surface waters.

Brief Summary Text (7):

Pond and lake <u>fertilization</u> has undergone several stages of development over time. Recently, these changes have resulted from attempts to reduce the rising cost of <u>fertilization</u> and to respond to concerns relating to the effects of <u>fertilizer</u> on the environment.

Brief Summary Text (8):

In early <u>fertilization</u> programs, granular N-P-K <u>fertilizers</u> such as 8-8-2 or 20-20-5 compositions were simply broadcast over shallow areas of ponds, or poured from a boat along the shallow water edges. Later research demonstrated that placing

fertilizers on an underwater platform produced similar results, required less fertilizer, and was less time-consuming. Wind and wave action were found to distribute nutrients throughout the pond and to make them more readily available to phytoplankton rather than tied up by pond muds. A single, well-placed platform could service a pond with up to 6 surface hectares (ha) of water. Subsequent findings indicated that ponds with a history of fertilization required only phosphorus fertilization and costs could be substantially reduced.

Brief Summary Text (9):

Despite the proven performance of <u>fertilizer</u> platforms, few pond owners used platforms in actual practice. Probably the biggest breakthrough in the use of fish pond <u>fertilizers</u> came in the form of liquid <u>fertilizers</u>. Besides the superiority of liquid <u>fertilizers</u> over granular <u>fertilizers</u> in increasing fish yields, liquid <u>fertilizers</u> had several other attractive attributes. Liquid <u>fertilizers</u> which are almost totally soluble in pond or lake water if properly applied could be used effectively at reduced application rates relative to prior granular products, were relatively easy and safe to apply, and were relatively economical to use.

Brief Summary Text (10):

Thus, it has been known previously to apply common, soluble <u>fertilizers</u> to ponds and lakes in order to increase inorganic nutrient concentrations, to favor greater phytoplankton growth and to ultimately enhance production of fish and/or crustaceans in the aquatic ecosystem. Current practice involves the use of liquid <u>fertilizers</u> or common, soluble granular <u>fertilizers</u> such as urea, ammonium, phosphates, ammonium polyphosphate, potassium sulfate and the like which are applied to closed aquatic ecosystems such as ponds and lakes throughout a production cycle. Frequent applications are required to keep the nutrients available in solution.

Brief Summary Text (11):

However, the prior art techniques have been unable to overcome problems presented by the need for constant availability of nutrients for growth of phytoplanktons and the fact that the previously proposed <u>fertilizer</u> products for use in aquaculture have provided nutrient availability for only a short duration of time after application. Accordingly, it has been necessary heretofore to apply such <u>fertilizers</u> up to 8-10 times per production cycle in order to maintain uniform phytoplankton growth within a given closed aquatic ecosystem such as a pond or a lake. If such an application schedule is not followed, it has been found that fish production in the environment suffers. Of course, such repeated application schedules are expensive, inefficient and time consuming.

Brief Summary Text (12):

Thus, the use of inorganic granular fertilizers in aquaculture has been considered to be costly and inefficient because of rapid dissolution of most fertilizer materials and leaching into the aquatic ecosystem and the inability to utilize nutrients efficiently.

Brief Summary Text (14):

Accordingly, it is a primary object of the present invention to provide compositions and methods which overcome the problems and deficiencies which have been encountered in regard to prior art fertilization techniques employed in aquaculture.

Brief Summary Text (15):

A further object is to provide methods for applying <u>fertilizers</u> to a closed aquatic ecosystem in a manner such that adequate nutrients are provided to support growth of phytoplanktons therein, over an extended period of time, without requiring multiple fertilizer applications.

Brief Summary Text (16):

Another object is to provide <u>fertilizer</u> products which may be applied in a single application to closed aquatic ecosystems such as ponds, lakes, watersheds and other aquatic environments and at lower dosage rates than previously utilized products in order to achieve satisfactory growth of phytoplankton populations in the environment over an extended period of time.

Brief Summary Text (17):

A further object is to provide a method and composition for effectively <u>fertilizing</u> fish ponds, lakes, watersheds and like aquatic environments in a more efficient and cost effective manner than has been accomplished heretofore.

Brief Summary Text (18):

The foregoing and other objects of this invention are achieved by providing controlled release <u>fertilizer</u> compositions which release nutrients at a controlled rate over an extended period of time for treating closed aquatic ecosystems such as ponds, lakes, watersheds and like aquatic environments. Such controlled release materials are formulated to delay or reduce the rate of nutrient delivery to the aquatic environment.

Brief Summary Text (19):

Controlled release <u>fertilizer</u> technology has been widely used heretofore in the agricultural and horticultural fields for a considerable amount of time and encompasses the controlled delivery of plant nutrients , i.e., <u>fertilizers</u>, as well as control chemicals (e.g., herbicides, insecticides, fungicides and the like) to a target in a manner which maximizes its efficient use, minimizes potential negative effects associated with overdosage, and/or extends the time in which sufficient dosages are delivered. However, such <u>fertilizer</u> products have not been employed heretofore in aquaculture.

Brief Summary Text (20):

Exemplary of the advantages attained in using controlled release <u>fertilizers</u> in aquaculture is that pond <u>fertilization</u> is simplified to a one-or-two step operation during each production cycle. Furthermore, controlled release <u>fertilizer</u> compositions can be used at low application rates and in pond systems with moderate water exchange. Further, controlled release <u>fertilizer</u> compositions are safe to use in pond waters and are readily available from commercial sources.

Brief Summary Text (21):

Accordingly, the methods of the present invention are based on the use of controlled release <u>fertilizers</u> to release nutrients into closed aquatic ecosystems such as ponds, lakes, watersheds and other aqueous environments in order to promote the growth of phytoplanktonic algae in the water in a more efficient and cost effective manner than has been accomplished employing previously known aquaculture <u>fertilization</u> techniques.

Brief Summary Text (23):

In general, the method of the present invention includes the application of a controlled release <u>fertilizer</u> into a closed aquatic ecosystem. The controlled release <u>fertilizer</u> is formed from a particulate core material having a release material reacted with or coated thereon. The release material is present in an amount sufficient to provide slow release of the core material into the surrounding aquatic environment in sufficient quantities to promote phytoplankton populations to support marine life in the aquatic ecosystem over an extended period of time.

Brief Summary Text (24):

Marine life which are supported by the phytoplankton algae populations in the aquatic environment include a wide variety of fresh water, brackish water and salt water fish as well as crustaceans such as shrimp, shellfish, crawfish and the like. The expression "marine life" as employed herein is intended to encompass all such forms of aquatic life. Thus, the use of controlled release <u>fertilizers</u> as nutrient delivery systems to enrich aquatic phytoplankton populations in aquaculture production in lakes and ponds has important implications from the standpoint of such enterprises as sport fish ponds; catfish, bait fish and crawfish production ponds; shrimp farm ponds; tropical fish production; exotic aquaculture such as alligator, eel and other novelty or high value crops; fish hatchery ponds and the like.

Brief Summary Text (25):

Suitable <u>fertilizer</u> compositions for use herein have been referred to in the agricultural and horticultural fields under various designations including controlled release, controlled availability, slow-release, slow acting, metered release, linear release and delayed release <u>fertilizers</u> (all of which are collectively referred to herein as "controlled release" <u>fertilizers</u>).

Brief Summary Text (26):
Controlled release <u>fertilizers</u> for use in this invention are granular <u>fertilizers</u> which may be prepared either as reaction products or as coated products. Examples of granular controlled release fertilizer reaction products for use herein are urea-formaldehyde (UF) reaction products such as ureaform, methylene ureas and MDU/DMTU compositions as described, for example, in U.S. Pat. No. 4,378,238, entitled: "Controlled Release Particulate Fertilizer Composition", the disclosure of which is incorporated herein by reference; urea-other aldehyde reaction products such as isobutylidene diurea (IBDU), guanyl ureas, and crotonylidene diurea (CDU); and other reaction products such as oxamide and melamine fertilizers, inorganic metal complexes such as magnesium-ammonium phosphate (magamp), magnesium-potassium phosphate and the like.

Brief Summary Text (27):

Coated controlled release fertilizers for use in accordance with this invention are products in which soluble fertilizer core granules (substrates) are covered with a water insoluble or semipermeable coating or release material which limits or controls the rate of water penetration to the soluble fertilizer cores, and controls the release rate of solubilized fertilizer from within the granules to the external environment. In preferred embodiments of the present invention the coating or release material is applied on the core material in an amount of about 0.5 to 35% (by weight).

Brief Summary Text (28):

Examples of coated granular controlled release fertilizers are those using sulfur as the coating material; those that employ a wax and/or a polymeric material and those hybrid products that utilize a multilayer coating of sulfur and polymer. Polymers suitable for use herein include polyvinyl chloride, polyvinylidene chloride, polyethylene, polypropylene, polyethylene terephthalate, polyurethane, polyamides, copolymers of dicyclopentadiene and vegetable oils such as linseed or soybean oil, copolymer blends of predominantly vinylidene chloride monomers and ethylenically unsaturated comonomers, salts of sulfonated elastomers and mixtures thereof. Furthermore, polymer coatings described in U.S. Pat. No. 4,657,576, entitled: "Granular Fertilizer Composition Having Controlled Release and Process for the Preparation Thereof"; U.S. Pat. No. 5,089,041, entitled: "Encapsulated Slow Release Fertilizers"; U.S. Pat. No. 5,300,135, entitled: "Abrasion Resistant Coatings for Fertilizers"; and U.S. Pat. No. 5,219,465, entitled "Sulfur Coated Fertilizers and Process for the Preparation Thereof" the disclosures of which are incorporated herein by reference, are particularly suited for use herein.

Brief Summary Text (29):

In addition, it is advantageous to incorporate oxidant materials such as peroxides, nitrates and mixtures thereof in the controlled release fertilizer compositions of this invention or to provide such oxidants in combination with the controlled release fertilizer compositions. Preferred oxidant materials for use herein include calcium peroxide, sodium peroxide, potassium peroxide, calcium nitrate, sodium nitrate, potassium nitrate, ammonium nitrate, magnesium nitrate and mixtures thereof.

Brief Summary Text (30):

Also, it is advantageous to formulate micronutrient materials such as iron, zinc, boron, calcium, magnesium, sulfur, manganese, copper, molybdenum, cobalt and mixtures thereof along with the nitrogen, phosphorus and/or potassium cores or the micronutrients can be formulated separately in the compositions of this invention. The micronutrients themselves may be formulated collectively or separately. It is particularly preferred to incorporate such micronutrients for purposes of aquaculture as chelates or salts in the form of nitrates, phosphates, oxides, chlorides, borates, molybdates, sulfates and mixtures thereof into or with the present compositions.

Brief Summary Text (31):

Aquaculture fertilizer production requirements vary widely depending on crop, water quality and environmental conditions. Typical suggested rates for nitrogen, phosphorus and potassium per production cycle are:

Brief Summary Text (35):

The <u>fertilizer</u> core materials are specifically formulated to release from one month to twelve months, depending upon the environmental and production parameters. Typical <u>fertilizer</u> core materials will be formulated to release for 8 to 9 months in the southern United States.

Detailed Description Text (4):

Box-like wooden platforms (interior dimensions 114 cm.times.60 cm.times.9 cm deep) for placement of controlled release fertilizer samples (referred to hereinafter as "CRF samples") and instant release, soluble granular fertilizer samples (referred to hereinafter as "granular fertilizer samples") were constructed and anchored to existing piers such that platforms were about 30 cm underwater. Granular fertilizer sample treatments were based on a "standard" application rate of 9 kg P.sub.2 O.sub.5 /ha and ten applications per season. Ponds were randomly divided into three treatment groups. One treatment group was a control in which granular fertilizer was applied as the fertilizer source. In the other two treatment groups, CRF fertilizer samples were employed at 100% and 50% application rates, respectively.

Detailed Description Text (5):

Nutrient sources for the soluble granular <u>fertilizer</u> samples were ammonium nitrate (33.5% N), triple superphosphate (46% P.sub.2 O.sub.5), and muriate of potash (60% K.sub.2 O). The nutrient constituents of the CRF samples were ammonium nitrate (33.5% N), ammonium phosphate (11-18% N, 48% P.sub.2 O.sub.5), and potassium sulfate (50% K.sub.2 O).

Detailed Description Text (7):

The Osmocote controlled release <u>fertilizers</u> used in this testing had a grade of 13-13-13 (%N, %P.sub.2 O.sub.5, %K.sub.2 O) and have been found to be particularly suitable for use in the present invention.

Detailed Description Text (8):

The controlled release fertilizer treated ponds received a one time 45 or 90 kg (N, P.sub.2 O.sub.5, K.sub.2 O)/ha application on March 21. The granular soluble fertilizer treated ponds received a 9 kg (N, P.sub.2 O.sub.5, K.sub.2 O)/ha application on March 21, and this was repeated on April 5, April 18, May 9, May 30, June 21, July 20, August 20, and September 20 to provide application rates in accordance with the data in the following Table 1:

Detailed Description Text (12):

Twelve 0.02-0.07 hectare (ha) aquaculture ponds were treated with CRF fertilizer samples for purposes of evaluation during a series of tests which were conducted. These tests were carried out under similar conditions to those described above with regard to Example 1 except that soluble liquid fertilizer samples replaced the granular samples of Example 1.

Detailed Description Text (14):

The nutrient source for the liquid <u>fertilizer</u> treatments was ammonium polyphosphate (10% N, 34% P.sub.2 O.sub.5). Application of the liquid <u>fertilizer</u> consisted of diluting it in a bucket of water and broadcasting it evenly over pond surfaces. <u>Fertilizers</u> were added to all ponds on April 20. The liquid <u>fertilizer</u> treatment ponds also received <u>fertilizer</u> applications on May 11, June 7, June 22, July 19, and August 16.

Detailed Description Text (15):

Despite <u>fertilization</u>, few ponds (2) had phytoplankton blooms. On May 20, a decision was made to systematically drain (80% volume), manually remove problematic weeds and algae, and refill all ponds. This work was done over a two week period. Because of this action, existing Osmocote <u>fertilizer</u> was removed and replaced in all controlled release treatment ponds on June 7. On June 9, all ponds were limed again (600 kg/ha).

Detailed Description Text (18):

The present studies have demonstrated that controlled release <u>fertilizers</u> can be used at much lower rates and give similar results in terms of water quality and fish

production as compared with prior art <u>fertilization</u> techniques. Furthermore, the use of the controlled release <u>fertilizers</u> in aquaculture enables the important commercial benefit of having to <u>fertilize</u> only once a season. Still further, controlled release <u>fertilizers</u> conserve valuable nutrient resources and work well in systems with moderate water exchange as demonstrated herein.

Detailed Description Paragraph Table (1): Amounts of fertilizer constituents per yearly season for three different treatments was replicated twice. Application rate (kg/ha per season) Treatment N P.sub.2 O.sub.5 K.sub.2 O Granular Fertilizer 90 90 90 Samples* 100% CRF Samples 90 90 90 50% CRF Samples 45 45 45 *This translates into 9 kg (N, P.sub.2 O.sub.5, K.sub.2 O)/ha per individual application. Detailed Description Paragraph Table (2): Summary of mean fish production values in ponds which received a standard dosage of granular fertilizer (13-13-13) and two levels of a controlled release fertilizer (13-13-13). Each treatment was replicated twice. Treatment Granular Fertilizer 100% CRF 50% CRF Measurement Samples Yield of bluegill (kg/ha) 95 Samples Samples 99 32 Yield of tilapia (kg/ha) 77 63 34 Yield of tilapia & bluegill 1256 1236 689 juveniles Yield of grass carp (hg/ha) 281 189 240 Total yield (kg/ha) 1709 1588 995 Average bluegill wt. (g) 23.5 26 22.5 % survival of original stock 69 63 22 Average tilapia wt. (g) 190 124 94.5 % survival of original stock 83 100 75 Average grass carp wt. (g) 1386 664 873 % survival 67 80 89 Detailed Description Paragraph Table (3): Summary of mean water quality TABLE 3 variables in ponds which received a standard dosage of a granular fertilizer (13,-13-13) and two levels of a controlled release fertilizer (13-13-13) during 1992. Each treatment was replicated twice. Treatment Granular Fertilizer 100% CRF 50% CRF Variable Samples Samples Samples 0.16 0.17 0.09 SRP (mg/L) 0.10 0.07 0.03 No.sub.3 --N (mg/L) 0.15 0.17 0.14 TAN (mg/L) 0.03 0.03 0.01 Chlorophyll a (.mu.g/l) 31 26 20 pH 9.0 8.9 8.8 Secchi visibility (cm) 56 54 60 Gross primary productivity 2.2 2.9 1.4 (mg O.sub.2 / 1/6 hr) Alkalinity (mg/L as CaCO.sub.3) 38 40 39 Hardness (mg/L as CaCO.sub.3) 39 41 39 Potassium (mg/L) 3.7 4.2 2.6 Detailed Description Paragraph Table (4): TABLE 4 Amounts of fertilizer constituents per season for four different treatments. Nutrients were applied 6 times between April 20 and September 13 using a liquid fertilizer sample. A one-time application of CRF sample was based on an application rate of 9 kg P.sub.2 O.sub.5 /ha and ten applications per season. Each treatment was replicated three times. Application rate (kg/ha per season) Treatment Grade N P.sub.2 O.sub.5 K.sub.2 O Liquid 10-34-0 26.5 90 0 Fertilizer Sample 50% CRF Sample 13-13-13 90 90 90 25% CRF Sample 13-13-13 22.5 22.5 22.5 12.5% CRF Sample 13-13-13 11.3 11.3 11.3 Detailed Description Paragraph Table (5): Summary of mean fish production values in ponds which received a standard dosage of a liquid fertilizer (10-34-0) and three levels of a controlled release fertilizer (13-13-13). Each treatment was replicated three times. Treatment Liquid CRF CRF CRF Measurement Fertilizer Sample Sample Sample Application Rate Sample (50%) (25%) (12%) Yield of bluegill (kg/ha) 90 99 93 86 Yield including juveniles 259 233 360 183 (kg/ha) Yield of grass carp 147 163 135 (kg/ha) Total yield (kg/ha) 408 397 495 272 Average bluegill wt. (g) 22.4 17.9 16.9 17.5 % survival 73 99 102 90 Est. juvenile (g) 0.81 0.76 0.77 0.93 Average grass carp wt.

Detailed Description Paragraph Table (6):

1405 617 677 466 (g) % survival 76 172 104 121

Summary of mean water quality variables in ponds which received a standard dosage of a liquid fertilizer (10-34-0) and three levels of a controlled release <u>fertilizer</u> (13-13-13). Each treatment was replicated three times. Treatment Liquid <u>Fertilizer</u> CRF CRF CRF Variable Sample Sample Sample Sample Application Rate (100%) (50%) (25%)

NH.sub.4 --N (mg/L) 0.02 0.07 0.02 0.02 NO.sub.2 --N (mg/L) 0.004 0.007 0.004 0.002 NO.sub.3 --N (mg/L) 0.23 0.35 0.23 0.16 SRP (mg/L) 0.06 0.03 0.02 0.01 TP (mg/L) 0.20 0.24 0.14 0.11 Chlorophyll a (.mu.g/l) 59 75 66 35 pH 8.9 9.1 9.0 8.7 Secchi visibility (cm) 57 49 57 60 Gross primary 2.4 3.2 2.8 2.1 productivity (mg O.sub.2 / 1/6 hr) Alkalinity (mg/L as 32 31 32 39 CaCO.sub.3) Hardness (mg/L as 35 34 35 44 CaCO.sub.3) Potassium (mg/L) 2.1 2.7 2.7 2.7 Weed coverage 42 21 21 18 (% of bottom)

Other Reference Publication (4):

Boyd, C. E. "Fertilization of Warmwater Fish Ponds". Journal of Soil and Water Conservation. 1981. 36:142-145. No Month.

Other Reference Publication (5):

Boyd, C. E. "Comparison of Five Fertilization Programs for Fish Ponds". Transactions of the American Fisheries Society. 1981. 110:541-545. No Month.

Other Reference Publication (6):

Boyd, C. E. "Liquid Fertilizers for Fish Ponds". Alabama Agricultural Experiment Station. 1982. 28:3-4. No Month.

Other Reference Publication (9):
Boyd, C. E. and J. R. Snow. "Fertilizing Farm Fish Ponds". Alabama Agricultural Experiment Station Auburn University. 1975. Leaf.88. 8 pp. No Month.

Other Reference Publication (10):

Boyd, C. E. and J. W. Sowles. "Nitrogen Fertilization of Ponds". Transactions of the American Fisheries Society. 1978. 107:737-741. No Month.

Other Reference Publication (11):

Hauck, R. D. and M. Koshino. "Slow-Release and Amended Fertilizers". Fertilizer Technology & Use. Madison, Wisconsin. 1971. 455-493. No Month.

Other Reference Publication (13):

Hepher, B. "I. The Effect of Fertilization on Fish Yields". Ten Years of Research in Fish Ponds Fertilization in Israel. 1962. Bamidgeh 14:29-38. No Month.

Other Reference Publication (14):

Hepher, B. "II. Fertilizers Dose and Frequency of Fertilization". Ten Years of Research in Fishpond Fertilization in Israel. 1963. Bamidgeh 15: 78-92. No Month.

Other Reference Publication (15):

Lawrence, J. M. "A New Method of Applying Inorganic Fertilizer to Farm Fishponds". The Progressive Fish-Culturist. 1954. 16:176-178. No Month.

Other Reference Publication (16):

Lunt, O. R., J. J. Oertli, and A. M. Kofranek. "Coated Fertilizers `Meter Out` Plant Nutrients". Crops & Soils. 1962. 14:14-15. No Month.

Other Reference Publication (17):

Metzger, R. J. and C. E. Boyd. "Liquid Ammonium Polyphosphate as a Fish Pond Fertilizer". Transactions of the American Fisheries Society. 1980. 109:563-570. No Month.

Other Reference Publication (18):

Mortimer, C. H. Fertilizers in Fishponds. Her Majesty's Stationary Office, London. 1954. Fish. Publ. No. 5. 155 pp. No Month.

Other Reference Publication (19):

Neess, J. C. "Development and Status of Pond Fertilization in Central Europe". Transactions of the American Fisheries Society. 1946. 76:355-358. No Month.

Other Reference Publication (20):

Nichols, W. C. "Application of Liquid <u>Fertilizer</u> to Hatchery Ponds". Progressive Fish-Culturist. 1983. 45:223-225. No Month.

Other Reference Publication (21):

Oertli, J. J. and O. R. Lunt. "Controlled Release of <u>Fertilizer Minerals by</u> Incapsulating Membranes: I. Factors Influencing the Rate of Release". Soil Science Society Proceedings. 1962. 26:579-583. No Month.

Other Reference Publication (22):

Parks, R. W., E. Scarsbrook, and C. E. Boyd. "Phytoplankton and Water Quality in a Fertilized Fish Pond". Agricultural Experiment Station Auburn University, Auburn, AL. Cir. 224. 16 pp. No Date.

Other Reference Publication (23):

Rabanal, H. R. "The Effect of No Fertilization and Non-Nitrogenous Fertilization Upon the Chemistry of Water, the Plankton, Bottom Organism and Fish Production in Ponds That Had Received Complete (N-P-K) Fertilizers for the Preceding 15-year Period". Ph.D. dissertation. Auburn University, Auburn, AL. 95 pp. No Date.

Other Reference Publication (24):

Reeves, W. C. and F. R. Harders. "Liquid Fertilization of Public Fishing Lakes in Alabama". Proc. Annu. Conf. SEAFWA. 1983. 37:371-375 No Month.

Other Reference Publication (26):

Smith, E. V. and H. S. Swingle. "Effect of Organic and Inorganic Fertilizers on Plankton Production and Bluegill Bream Carrying Capacity of Ponds". Transactions of the American Fisheries Society. 1939. 69:257-263. No Month.

Other Reference Publication (27): .

Smith, E. V. and H. S. Swingle. "The Use of Fertilizer for Controlling Several Submerged aquatic Plants in Ponds". Transactions of the American Fisheries Society. 1941. 71:94-101. No Month.

Other Reference Publication (28):

Swingle, H. S. "Fertilizing Farm Fish Ponds". Agricultural Experiment Station of Auburn University. 1965. Res. 12:11. No Month.

Other Reference Publication (29):

Swingle, H. S. and E. V. Smith. "Fertilizers for Increasing the Natural Food for Fish in Ponds". Transactions of the American Fisheries Society. 1939. 68:126-135. No Month.

Other Reference Publication (31):

Swingle, H. S. "Experiments on Pond Fertilization". Alabama Agricultural Experiment Station Bull. 264, Auburn, AL. 1947b. 36 pp. No Month.

Other Reference Publication (32):

Swingle, H. S., B. C. Gooch, and H. R. Rabanal. "Phosphate <u>Fertilization</u> of Ponds". Annual Conference of the Southeastern Association of Game and Fish Commissioners. 1963. 17:213-218. No Month.

Other Reference Publication (33):

Wiebe, A. H. "The Effects of Various <u>Fertilizers</u> on Plankton Production". Transactions of the American Fisheries Society. 1929. 59:94-105. No Month.

Other Reference Publication (34):

Dobbins, D. A. and C. E. Boyd. "Phosphorus and Potassium Fertilization of Sunfish Ponds". Transactions of the American Fisheries Society. 1976. 105:536-540. No Month.

CLAIMS:

1. A method for promoting marine life populations in a closed aquatic ecosystem

comprising applying a controlled release <u>fertilizer</u> composition into said ecosystem, said controlled release <u>fertilizer</u> composition being formed from a particulate core material and a release material reacted with or coated on said core material, said core material being selected from the group consisting of phosphorus, nitrogen and potassium sources and mixtures thereof and said controlled release <u>fertilizer</u> composition being formulated to provide slow release of a sufficient amount of said core material from said release material into said closed aquatic ecosystem, in a single application of said controlled release <u>fertilizer</u> composition thereto, to promote stable growth of phytoplankton algae in said ecosystem without causing overly dense growth of said phytoplankton algae, whereby marine life populations in said closed aquatic ecosystem are maintained over an extended period of time.

- 10. The method of claim 1 wherein an oxidant material is included with said controlled release <u>fertilizer</u> composition, said oxidant material being selected from the group consisting of peroxides, nitrates and mixtures thereof.
- 12. The method of claim 1 wherein a <u>micronutrient</u> material is included with said controlled release <u>fertilizer</u> composition, said <u>micronutrient</u> material being selected from the group consisting of iron, zinc, boron, calcium, magnesium, sulfur, manganese, copper, molybdenum, cobalt and mixtures thereof.
- 13. The method of claim 12 wherein said $\frac{\text{micronutrient}}{\text{of nitrates}}$ material is a chelate or a salt selected from the group consisting $\frac{\text{micronutrient}}{\text{of nitrates}}$, phosphates, oxides, chlorides, borates, molybdates, sulfates and mixtures thereof.

Generate Collection Print

L12: Entry 6 of 10 File: USPT Apr 26, 1977

DOCUMENT-IDENTIFIER: US 4019890 A

TITLE: Method for producing coated fertilizer

Assignee Name (1):

Chissoasahi Fertilizer Co., Ltd.

Assignee Group (1):

Chissoasahi Fertilizer Co., Ltd. Tokyoto JA 03

Abstract Text (1):

Granular slow-releasing fertilizers completely coated with water-resisting resin are produced in such a manner that the coated granules do not stick together, through one step operation by spraying a solution of the resin which has the property capable of maintaining the solution state while it is hot but turning into jelly-like gel when it is cooled, on granules of fertilizer and drying the said granules with a high speed drying hot air on the spot upon being sprayed with the solution of the resin.

Brief Summary Text (2):

From the requirement of the modernization of farming, <u>fertilizers</u> which have properties suitable for mechanization and labour saving have been sought for. As <u>fertilizers</u> capable of meeting the above-mentioned purpose, liquid <u>fertilizers</u> and slow-releasing fertilizers have been developed and are appearing in the market.

Brief Summary Text (3):

The fertilizers which have been developed as slow-releasing fertilizers can be roughly classified into following three kinds; (1) fertilizers whose components have been turned to give difficultly soluble or difficultly decomposable properties through a chemical way, (2) fertilizers which are turned into difficultly soluble form by some methods for example incorporation of inorganic substances such as cements or the like or organic substances such as wax or the like and (3) fertilizers which are coated with a water-resisting substance.

Brief Summary Text (4):

The fertilizers having the form of (1) include organic nitrogen-containing compounds such as crotylidene diurea (CDU) obtained by the condensation of urea and aldehyde, isobutylidene diurea (IBDU), urea form (UF), oxamide, etc. and difficultly soluble salts such as ammonium magnesium phosphates, etc. The release of effective component of this form of fertilizer is influenced a great deal by soil microorganisms, soil water and soil pH and hence the degree of release varies according to kind and water content of soil. So it is generally difficult to control the dissolution in accordance with the requirement of plants.

Brief Summary Text (5):

The <u>fertilizers</u> having forms (2) and (3) have advantage in that the <u>fertilizers</u> which have been heretofore used widely can be used as raw materials as they are. Although the dissolution control of the <u>fertilizer</u> having the form (2) is carried out by varying the kind and addition amount of solidifying binder, finding of a suitable binder is rather difficult under the condition that it must be cheap and available in a large amount. Moreover the form (2) has a drawback in the point that dissolution control cannot be performed freely due to its inherent property. The <u>fertilizers</u> of the form (3) may have the possibility of being more freely controlled

in dissolution, but if coating is not perfect and contains even a slight extent of pinholes, water-soluble components dissolve out within a short time and if coating is perfect on the contrary, dissolution does not take place, etc. Thus, the fertilizers of the form (3) still have problems which have not been solved yet in the control of dissolving rate.

Brief Summary Text (6):

As the methods for producing coated <u>fertilizers</u> which have found their practical application, a multiple layer coating method which uses a thermosetting resin and sulfur coating method which utilizes molten sulfur can be mentioned. Although in the former case the control of dissolving rate is made by the pinholes created during the process of production and remaining thereafter and in the latter case, by incorporating paraffin or the like and allowing the coating to be decomposed by soil microorganisms, optional control is not possible in both the cases.

Brief Summary Text (7):

In order to control the dissolving property of coated <u>fertilizers</u> freely, a perfect coating should be made because fine pinholes can be made for the purpose of dissolving control relatively easily if perfect coating with uniform, very thin film is once made. In order to provide perfect coating for controlling dissolving property, it is necessary to dissolve in a solvent a coating material which is superior in resistance to water, moisture-impermeability and resistance to osmotic pressure, and to effect coating in the form of solution. However, the maximum defect of the solution method is the mutual agglomeration of <u>fertilizer</u> granules during coating treatment and on this account, it has been regarded that individual granules cannot be coated by the solution method.

Brief Summary Text (8):

Accordingly, it is an object of the present invention to provide a method for producing completely coated granular <u>fertilizer</u>, using a solution of highly water-resistant resin, in such a manner that the coated granules do not stick together and through one step.

Brief Summary Text (9):

It is another object of the present invention to provide a method for producing granular <u>fertilizers</u> completely coated with resin films wherein, however, control of dissolution of <u>fertilizer</u> component can be made by adding a necessary additive for forming pinholes.

Brief Summary Text (10):

These objects and other advantages can be attained by the method of the present invention. The present invention lies in a method for producing slow releasing granular fertilizer fully coated with water-resisting, transparent, lustrous layer of resin in such a manner that the coated granules do not stick together, which comprises spraying a solution of a thermoplastic resin in a hydrocarbon or chlorinated hydrocarbon solvent, upon fertilizer granules, and drying said fertilizer granules upon being sprayed with said solution, continuously and immediately on the spot with a high speed hot air, said solution having the property of capable of forming a solution while it is hot but turning into jelly-like gel when it is cooled.

Brief Summary Text (12):

The resin solutions used in the method of the present invention are those obtained from a combination of resin and a solvent in which a solution is formed while it is hot but resin deposits and turns into jelly-like gel when it is cold. A representative example of this case is a solution consisting of a polyolefin and a solvent of petroleum hydrocarbon or chlorinated hydrocarbon and this is a most preferable solution for the present invention. It has been anticipated that this kind of solution would be entirely inadequate for the coating of fertilizer granules because the coating carried out by using this solution would bring about deposit and gelation of resin component on the surface of particles during coating step and hence no film is formed. However, it has been found that even when the coating of individual granules is carried out by using the solution having the property of forming jelly-like gel when it is cooled (hereinafter referred to a gel-forming solution), there is an advantage that any sticking or agglomeration which has been a

problem in the past does not occur.

Brief Summary Text (13):

In the operation of coating of granular <u>fertilizers</u>, the <u>fertilizer</u> particles are usually mixed by rolling motion in a revolving drum, a revolving pan or the like or by fluidization in a fluidized bed and a liquefied coating material is added in spray form. If the liquefied coating material is a molten substance, solidification of granule surface is carried out by cooling and if it is a solution, drying is carried out by hot wind. During this course of step, a common solution is concentrated and increase of stickiness and agglomeration appear. In case of gel-forming solution, gelation temperature is elevated with the increase of concentration, and gelation always occurs irrespective of temperature, and film-form coating is not attained by common drying. Capsules formed by common drying are nothing but piled up resin powders containing some amount of solvent and hence it is impossible to suppress the dissolution of <u>fertilizer</u> practically even when a ratio of coating is increased. When the movement of granules becomes vigorous, the once-adhered coating separates by the collision of granules to each other.

Brief Summary Text (14):

The gelation due to phase separation in solution seems to occur within an infinitesimal time in case of coating with a gel-forming resin solution but there is always a certain time allowance until gel is actually formed and if drying is performed instantaneously within this infinitesimal time, granular fertilizers can be encapsulated with a dense, uniform film having a small moisture-permeability, and granular fertilizers practically close to perfect coating can be obtained without forming agglomeration.

Brief Summary Text (15):

The characteristic effectiveness of the method of the present invention lies in that no agglomeration occurs even when a very large amount of solution is added within a short time and efficient coating of individual granules can be made. This effectiveness of the present invention is attained by adding a resin solution having a high gelation property in spray form to fertilizer granules while the solution is maintained at a temperature higher than the phase separation temperature and drying the granules with a high speed stream of hot wind for drying, continuously and immediately on the spot hit with spray.

Brief Summary Text (17):

The most important conditions in the production according to the method of the present invention are stream speed of hot wind and temperature of granules. Stream speed of hot wind should be 5 m/sec. or higher, preferably 15 m/sec. or higher on the spot or zone (or in situ) of <u>fertilizer</u> granules sprayed with said solutions. The higher the speed, the better the result. A suitable temperature of granules varies according to the properties of solution, and in cases of the solutions of polyolefins or polyvinylidene chloride in hydrocarbon or chlorinated hydrocarbon solvents which easily form gel and are preferable in the practice of the present invention, a temperature in the range of 40.degree.-90.degree. C, particularly 60.degree.-70.degree. C is preferable. If a temperature is lower than 30.degree.-40.degree. C, gelation occurs and dense transparent film coating cannot be obtained. In general, when a solvent having a boiling point higher than the gelation point of the solution by 60.degree.-80.degree. C is used, it is preferable to keep the temperature of granules in the range of temperature higher than the gelation point by 10.degree.-30.degree. C. However, when the boiling point of solvent and the gelation point of solution are close to each other, it is possible to use a lower temperature than the above-mentioned. To be strict since a latent heat of vaporization of solvent has influence, some correction may be necessary in the practice.

Brief Summary Text (21):

When the present invention is put into practice commercially, an easy solvent recovery is necessary, and for that purpose it is necessary to evaporate a large amount of solvent by using as small an amount of hot wind as possible. Thus a high speed stream of hot wind for drying should be concentrated onto the principal drying part and a resin solution should be added thereto. In this regard a spouted bed is a most suitable apparatus in the practice of the present invention. A spouted bed has

limiting conditions in the diameter of the cylindrical section of spouted bed, the diameter of gas inlet orifice for forming spout, the diameter of granules, the density of granules, the amount of spouting fluid, etc. Thus it is a drawback of this apparatus that optional conditions cannot be selected, but it will be sufficient if a wind speed of 15-40 m/sec. is selected as a speed of hot wind stream of the principal drying part for fertilizer granules having size of 2-5 mm in the spouted bed of the method of the present invention. If a solution of polyolefin or the like in tetrachloroethylene which forms jelly-like gel easily is treated under the above-mentioned conditions and apparatus, it is possible to evaporate 300-400 g of the solvent per 1 kg of air and there is no need of providing a subsequent drying step due to decreasing rate. In the method of the present invention, it is possible to select combinations from various resins and various solvents under the condition that gel-forming property is maintained. However, if a temperature of fertilizer granules during the operation of coating is too high, this becomes the origin for inducing various troubles such as thermal decomposition of fertilizer component, melt-adhesion of resin attached to the fertilizer, or the like, while if a temperature is too low, coating time is too prolonged and a lower boiling solvent is wastefully required, and hence unless granules contain a substance particularly weak to heat, such a lower temperature is not preferable. On this account it is advisable to use a gel-forming solution which can effect coating by maintaining the granule temperature during the coating operation at 40.degree.-90.degree. C, preferably 60.degree.-80.degree. C, in the method of the present invention. Thus it is preferable to select a solution having a gelation temperature of 30.degree. C or higher, preferably 50.degree. C or higher, in the case of 5% resin solution and a solvent having a boiling point in the range of 90.degree.-150.degree. C. A gelation temperature lower than 30.degree. C is not preferable because it causes agglomeration of granules during coating operation, and a lower boiling point solvent having a boiling point lower than the phase separation temperature is not useful because preparation of solution cannot be made under the atmospheric pressure and dissolution must be made under an elevated pressure. Thus it is preferable to use a solvent whose boiling point is higher than the phase separation temperature, by 20.degree. C or higher, preferably more than 40.degree. C or higher. However, if the boiling point of solvent is too high, efficient preparation is not possible and there are often cases where evaporation velocity becomes too small and gelated films cannot be obtained even according to the present invention.

Brief Summary Text (27):

The granular <u>fertilizers</u> used in the method of the present invention can have any component and granule size, but it is necessary to change the coating percentage according to granule size. Most preferable granule size is 3-5 mm in diameter. Granules smaller than this size are not preferable because coating percentage must be elevated and those larger than this size are also not preferable because non-uniformity of distribution is liable to be formed place to place when applied to forming fields. Coating treatment is in general easy in case of larger granule. As for granule shape, there is no need of being spherical but smooth surface is preferable.

Brief Summary Text (29):

It is easy to judge by appearance the difference of the capsule of the present invention from the capsule formed by common drying procedure of the solution used in the method of the present invention. The capsule obtained according to the method of the present invention has transparency and lustre same as those of common films and also a strong tensile strength, but the above-mentioned capsule formed by common drying shows white turbidity, and cannot be peeled off as film piece from the fertilizer granules, and moreover it has no lustre. Since it is nothing but piled up resin powders containing some amount of solvent, it has no sufficient mechanical strength. If granules collide strongly with each other during coating step, once-adhered coating is detached.

Brief Summary Text (30):

In the method of the present invention, measurement of properties of capsules is difficult and hence is not made for the purpose of evaluation thereof. Dissolving-out percent is measured and substituted for the above-mentioned purpose. This is because of the fact that to whatever the coating percent of gelated capsule is increased, practical effectiveness as fertilizer having slow releasing property

cannot be obtained and dissolving-out percent of capsule during the time of 24 hours is more than 90%, whereas the capsule obtained according to the method of the present invention shows only several % of dissolving-out percent.

Brief Summary Text (32):

The valuableness of the present invention is in the capability of controlling the dissolving rate of product. In the conventional methods such as a multiple coating method which uses a thermosetting resin or sulfur coating method, the release of fertilizer component has been controlled by providing pinholes in the coating of granules formed during preparation or by decomposition of sulfur by soil . microorganisms. However, in the method of the present invention, the control of dissolving is carried out by providing practically insoluble granular fertilizers completely coated with very thin film in the first step and by adding a certain additive e.g. a hydrophilic substance in the coating films to give an arbitrary dissolving speed in the second place.

Brief Summary Text (33):

Even when water-soluble fertilizers are completely coated for the first step object, it is not possible to stop the dissolution completely, because any films themselves have permeability to moisture and when fertilizers are exposed to water or the atmosphere of high steam pressure for a long time, water enters the inside of the capsule through the films, causes the deliquescense of the inside salts and elevates the osmotic pressure. However in case of a polyolefin having an extremely small moisture-permeability excepting armorphous polyolefin, dissolution is extremely small and practically neglibile if time is about one year. In case of polyvinylidene chloride having further smaller moisture-permeability, dissolution can be completely negligible. At any rate the first step object can be substantially attained.

Brief Summary Text (35):

If the main thermoplastic resin of the present invention is used in admixture with another thermoplastic resin having a higher moisture-permeability than that of the main resins, the resultant product shows, in general, a mean moisture-permeability of the two, whereby the dissolving speed of fertilizers is elevated. Said another thermoplastic resin is that soluble in solvents to be employed in the present invention and the amount thereof added is preferably 30% by weight or less. By utilizing said another thermoplastic resin having a higher moisture-permeability, dissolving speed of fertilizer components can be controlled as in the case of said surfactants. Further, said another thermoplastic resin is also useful as an extender of the main resins. Further, as materials to be added to polyolefins, waxes such as paraffin wax, hardened oil, etc. which are easily available in commerce can be mentioned. It is practical to add waxes in an amount of 30% by weight or less. When fertilizers are encapsulated with a resin containing a substances easily decomposable by soil microorganism such as paraffin wax or the like, the easily decomposable substance is decomposed and increases the dissolving speed.

Brief Summary Text (44):

3.0 kg of granules of Sun Nitro No. 1 (potassium ammonium nitrate phosphate complex fertilizer, N:P.sub.2 O.sub.5 :K.sub.2 O = 15:15:12, size 3.0-4.0 mm, product manufactured by Asahi Kasei Kogyo Kabushiki Kaisha, Japan) was introduced into a rotary pan having a diameter of 30 cm (sugar-coating machine), and then 5% (by weight) solution of polyethylene M6545 (MI 45) (trade mark of product manufactured by Asahi Dow Co., Ltd., Japan) in tetrachloroethylene, maintained at 100.degree. C was added to the granules in the form of atomized spray by means of a two-fluids-nozzle (a commercially available spray gun for coating), while the pan was rotated at 10 rpm and the temperature of the granules was maintained at given temperatures with a 3 M.sup.3 /min. hot air for heating, at 100.degree. C. The coating treatment was carried out under 5 kinds of temperature condition, i.e., the temperature was maintained at 40.degree.-50.degree. C, 50.degree.-60.degree. C, 60.degree.-70.degree. C 70.degree.-80.degree. C and 80.degree.-90.degree. C, respectively. In the procedure of preparation, 6.6 kg of the solution (containing 0.33 kg of polyethylene) was dried with hot air while the solution was added when the temperature reached the upper limit of the given range of temperatures, but the addition was stopped when the temperature fell to the lower limit thereof. Such drying and temperature elevation were repeated to complete the addition of the given amount of the solution. The granules thus treated got an increase of 0.3 kg.

Brief Summary Text (68):

4.0 kg of the granular <u>fertilizer</u> employed in Example 1 was introduced into the fluidized bed apparatus having a diameter of 25 cm and an inclination of 50.degree. at the conical part.

Brief Summary Text (69):

Coating treatment was carried out by rolling the granules under a plate-revolving of 200 rpm and drying them with hot air sent through the multi-perforated plate while adding a polyolefin solution thereto in a spray form through the nozzle part (a commercially available spray gun). The pipe for feeding the solution was insulated, and care was taken so that the temperature did not lower by 10.degree. C or more above the boiling point of the solvent at the nozzle part. Five % by weight solution of polyethylene (No. M 6545, trademark of product manufactured by Asahi Dow Co., Ltd., MI: 45) in tetrachloroethylene was added to the above-mentioned rolled and fluidized granules in a spray form, at a rate of 150 g/min, and under combinations of three kinds of space column velocities of hot air passed through the multi-perforated plate, in the fluidized bed, i.e., 0.5, 1.0 and 2.0 m/sec with 5 kinds of granule temperatures, i.e., 50.degree., 60.degree., 70.degree., 80.degree. and 90.degree. C. The temperature of hot air for drying in this case (T.sub. 1 in FIG. 1) was set to a temperature 3.degree.-4.degree. C higher than the granule temperature, although it is varied depending upon the amount of the hot air and the granule temperature. The solution was fed for 55 minutes and thereafter the granules were allowed to stand for 10 minutes and then taken out to give a coated granular fertilizer having a coating percent of 10% by weight.

Brief Summary Text (70):

The coated granular <u>fertilizer</u> obtained according to this treatment did not form a jellylike gel due to phase separation as in the case of Example 2, and some protruded parts of the capsules were peeled off by the rolling motion. Thus, under the drying condition of a space column velocity of 2 m/sec, no uniform film of capsules could be obtained. The nitrogen-dissolving out percent was observed under the same condition as in Example 2. A result after 24 hours was in the range of 90-98%. Thus, no practical effectiveness of encapsulation could be obtained.

Brief Summary Text (71):

Further, even when various polyolefins (excluding amorphous polypropylene) and various solvents were used as in Example 2, similar results were obtained. Thus, at a space column velocity lower than 2 m/sec, no slow-releasing coated granular fertilizer could be obtained. However, no agglomeration of granules due to mutual adhesion thereof was observed as in Example 2.

Brief Summary Text (78):

In order to further increase the velocity of air, the rotating multi-perforated plate part in FIG. 1 was replaced by a common multi-perforated plate (hole diameter: 3 mm, hole pitch: 5 mm, holes were arranged to form a right triangle) to form a usual fluidized bed. Fertilizer granules (the same as those employed in Example 2: 4 kg fed) were fluidized with hot air stream for drying, setting the space column velocity of air in the fluidized bed to 5 m/sec. A one-fluid-nozzle (nozzle opening: 0.6 mm) capable of forming a spraying angle of 60.degree. to the surface of the upper part of the fluidized bed was fixed at such a position that polyolefin solution could be sprayed over the whole surface of the fluidized bed. Thus, polyolefin solution in dissolved state was added at a rate of 300 g/min to carry out coating treatment. When the temperature of hot air below the multi-perforated plate, was set to a temperature about 10.degree. C higher than the granule temperature, said temperature of hot air came close to the set temperature of granules, and also the temperature of discharged gas (thermometer of discharged gas: T.sub.3 in FIG. 1) became almost equal to the granule temperature.

Brief Summary Text (80):

When other kinds of polyethylene (M-6520 and M-7620, both, products of Asahi Dow) were tested, similar results were obtained. According to this method, sprayed liquid drop was hardly attached onto the granular <u>fertilizer</u>, and polyolefin powder from which solvent vaporized off was existent in discharged gas in admixture therewith in a considerable amount, and also the amount of the powder attached onto the apparatus

was large, whereby the coating efficiency was reduced. Since such an apparatus was not desirable as mentioned above, further studies were made using an apparatus wherein the central part of the multi-perforated plate was opened to change the fluidized bed to a forced circulation type fluidized bed and a nozzle was provided at the central part of the resulting opening.

Brief Summary Text (92):

3. Hopper inlet of fertilizer granules

Brief Summary Text (104):

The conical part of the body has an inclination of 50.degree.. In order to make the movement of granules of fixed bed easy, a vibrator is provided. More particularly, the multi-perforated plate used above was removed, and instead, a teflon-coated steel pipe having a diameter of 10 cm and a length of 1 m was provided at the central part of the apparatus for the fluidized bed, in parallel to the body of the apparatus (i.e., vertically), so that the lower end of the pipe might be 5 cm apart from the wall of the conical part of the apparatus, and further a one-fluid-nozzle (spray angle: 30.degree.) 11 was provided vertically to the apex of the conical part (i.e., directed toward the center of the pipe at the central part). 4.0 kg of the fertilizer granules used in Example 2 was fed while hot air stream for drying was passed through within the cylinder. The fertilizer granules were carried and moved upwards within the cylinder provided inside the apparatus, by hot air stream, and during the course of this operation, they were coated. When they came out of the cylinder, the velocity was reduced below the terminal velocity, and they dropped onto the body side of the apparatus. The dropped granules formed a fixed layer, and gradually moved downwards to form a moving layer again and move upwards. In this Example, the amount of hot air was controlled so that the flow rate within the cylinder might be 15 m/sec (7.5 m.sup.3 /min) or 30 m/sec (15 m.sup.3 /min) and also the temperature of the granules in the solid phase was controlled to a given temperature, while the following solution was fed through a one-fluid-nozzle (opening: 0.6 mm), at a rate of 150 g/min for 55 minutes in the case of 15 m/sec, and at a rate of 300 g/min for 27 minutes in the case of 30 m/sec to give a fertilizer coated by 10% by weight of polyethylene. The temperature of the drying gas is to be set to a temperature about 10.degree. C higher than the granule temperature, but since it varies depending upon room temperature, humidity, etc, it was confirmed at each case.

Brief Summary Text (112):

3. Hopper inlet of fertilizer

Brief Summary Text (119):

10. Granular fertilizer (5 kg) 11. Solution tank

Brief Summary Text (121):

Hot air stream for drying was ejected at a rate of 4 m.sup.3 /min. 5.0 kg of granular fertilizer used in Example 2 was fed and a spout was formed, while polyethylene solution (obtained by dissolving M 6545 of Asahi Dow's product in tetrachloroethylene in 20 times the amount of weight of the polyethylene) was sprayed from the nozzle, to carry out coating treatment. As for the flow rate of the gas column in this case, the value of measurement, although not accurate, was about 15-20 m/sec (by means of Pitot tube). Under such conditions, the solution was added at a rate of 500 g/min. The temperature of hot air for drying was set to a temperature 30.degree. C higher than the granule temperature, and operation was started. During the operation, a certain extent of temperature correction was made to maintain the set temperature. The solution was fed for 20 minutes to give a coated granular fertilizer having a coating percent of 10%. The nitrogen-dissolving out percents after 24 hours are shown in Table 6. There was no great influence of granule temperature.

Brief Summary Text (125):

Using the same apparatus as in Example 8, 5 kg of the granular fertilizer used in Example 2 was subjected to spouted bed of hot air at a flow rate of 4 m.sup.3 /min. After the granule temperature reached 60.degree. C, various polyethylene solutions shown in Table 7 were fed for 10 minutes at a rate of 500 g/min to give products having coating percents shown in Table 7. Under such conditions, a certain extent of

adjustment was necessary for maintaining the temperature of the spouting air for drying to about 90.degree. C and also for maintaining the granule temperature during the coating treatment to 60.degree. C. The resulting products had no block and the treatment was well operated. The dissolving out percents in water after 24 hours, of the resulting coated granular <u>fertilizer</u> were measured. The results are shown in Table 7. As seen from these results, no difference due to M.I. was observed, but when the coating percent was lowered as in this Example, influence of resin density appeared, namely, a higher effectiveness of treatment was obtained with a higher resin density.

Brief Summary Text (128):

Under the same conditions of spouted bed as in Example 9, the effect of addition of waxes was observed. The granular <u>fertilizer</u>, amount of air, feeding rate of solution and temperature condition are same as those in Example 9. The conditions and the results are shown in Table 8.

Brief Summary Text (134):

Under the same conditions as in Example 9, coating treatment was carried out using the same granular <u>fertilizer</u> as in Example 2 and 5% by weight solution of polyethylene (No. M 6545 of Asahi Dow, M.I. 45) in tetrachloroethylene, while the granule temperature was maintained at the values given in Table 9, to give coated products. The nitrogen-dissolving out percents after 24 hours and the appearances of the products are measured as shown in Table 9.

Brief Summary Text (140):

5.0 kg of the <u>fertilizer</u> granules used in Example 2 was fed to the apparatus used in Example 8, and spout at a flow rate of 5 m.sup.3 /min was formed, as in Example 9. When the granule temperature reached 60.degree. C, resin solution was fed at a rate of 500 g/min to give a product having a coating percent of 4%. Tetrachloroethylene was used as a solvent. The resin employed, concentration, coating percent, solution-feeding time and coating effect are shown in Table 10. Other operation conditions are same as those of other Examples using spouted bed.

Brief Summary Text (145):

Coating of granular fertilizer was carried out using polyethylene M 7620 (M.I. 20) (manufactured by Asahi Dow), under the same conditions as in Example 9. Namely, the resin was dissolved in the solvents shown in Table 10 to prepare 3% by weight solution, which was then fed for 10 minutes at a rate of 500 g/min to give a coated granular fertilizer having a coating percent of 3%. During this preparation, the temperature of hot air was adjusted so as to maintain the granule temperature to 60.degree. C. The nitrogen-dissolving out percent in water, of the resulting coated fertilizers in this Example are shown in Table 11. As evident from this Table, any of the solvents employed give a highly water-resistant, coated granular fertilizer.

Brief Summary Text (153):

3.0 kg of the granular <u>fertilizer</u> used in Example 2 was fed in a rotating coating machine. Ten % by weight solution of amorphous polypropylene in xylene, described in the Example, was added to the granular <u>fertilizer</u> under rolling, by means of a spray gun, intermittently over one hour, and during this period, it was dried with hot air and the surface temperature of the granules was maintained at 70.degree.-80.degree. C. During this period, 120 g of amorphous polypropylene and 1.08 kg of xylene was added to give 3.120 kg. of coated fertilizer.

Brief Summary Text (155):

Using the apparatus described in Example 8, 5 kg of the above-mentioned granular fertilizer was subjected to spout of hot air at a flow rate of 4 m.sup.3 /min, while the above-mentioned solution of amorphous polypropylene in xylene was fed for 4 minutes at a rate of 500 g/min to give 5.2 kg of coated granular fertilizer. During this time, the granule temperature was maintained at 70.degree. C, and the temperature of hot air was in the vicinity of 115.degree. C. The nitrogen-dissolving out percents in water, of the resulting coated granular fertilizer were measured. The results are shown in Table 13.

Brief Summary Text (160):

In a spouted bed column having a shape similar to the apparatus indicated in FIG. 3,

a column diameter of 500 mm, a conical angle of 50.degree., a spout orifice of 100 mm, air of 15 m.sup.3 /min heated at 120.degree. C was passed as spout and 50 kg of granular fertilizer used in Example 2 was charged. A 3% by weight solution of polyethylene resin (supplier Asahi Dow Co. brand: M-7620) in tetrachloroethylene was passed through a one-fluid-nozzle (opening diameter of 2 mm) provided at the conical part of the spouted bed apparatus at a rate of 3.5 kg/min for 15 minutes while it was maintained at 100.degree. C. After completion of charging, the hot air was switched over to cold air and cooling was continued for 5 minutes under this condition, resulting in 51.5 kg of coated granular fertilizer. The temperature of granules at the time of feeding of the solution, and that of exhaust were maintained at 60.degree. C. The nitrogen-dissolving out percentage of the coated granular fertilizer for 24 hours was 0.5%. Thus it is evident that even when the scale of apparatus is enlarged, those having a high effectiveness of treatment are obtained.

Brief Summary Text (163):

Coated granular <u>fertilizer</u> was prepared by varying the size of granule used in Example 2 and varying the solution-feeding time under the same conditions as in Example 16, to observe the effectiveness of coating treatment. The results are shown in Table 14. The resin, solvent and concentration employed in this Example are same as those in Example 16.

Brief Summary Text (167):

Using the same apparatus as in Example 8, 5 kg of granular fertilizer used in Example 2 was spouted by the stream of hot air at a flow rate of 4 m.sup.3 /min, and after the granule temperature reached 60.degree. C, gel-forming solutions shown in Table 15 were fed at a rate of 500 g/min, for 10 minutes to give products having coating percent shown in Table 15. Under these conditions, the temperature of drying air for spout was maintained at 90.degree. C, and the granule temperature was maintained at about 60.degree. C. The nitrogen-dissolving out percents and extent of agglomeration of granules are also shown in Table 15. As evident from this Example, coating treatment can be readily carried out even with gel-forming solutions other than those of polyolefin.

Brief Summary Text (168):

Experiment No. 4 in Table 15 is an example where the temperature of drying hot air for spout was kept at 55.degree. C, and the feed rate of the solution was reduced to 250 g/min (fed for 20 minutes) to carry out coating. Under these conditions, no agglomeration of granules occurred, and coating treatment was possible. With resins containing 15% by weight or more of vinyl acetate, no gel-forming property was exhibited in any cases, and whatever conditions were set, granules were agglomerated during coating, spout stopped and encapsulation of individual granule could not be made. Further, as for the adjustment of solubility by means of surfactants, etc., shown in the Reference Example mentioned below, polyvinylidene chloride has not sufficient degree of freedom in adjustment. In this regard, polyolefins are preferable material for coating fertilizer granules.

Brief Summary Text (171):

Coated granular <u>fertilizer</u> was prepared under the same conditions as in Example 16 except that a polyethylene resin solution containing 0-15% of a nonioic surfactant (an octaoxyethylene nonylphenyl ether, ##STR1## based on the weight of the resin contained in the solution, was used. Test of dissolving out of <u>fertilizer</u> into water, of the product thus obtained was carried out to compare the nitrogen-dissolving out percents. The results are shown in FIG. 4.

Brief Summary Text (175):

A similar coating treatment to that of Example 9 was carried out by using a solution of a resin readily soluble in a solvent and highly water-resistant. Namely, 5.0 kg of granular fertilizer used in Example 2 was fed and subjected to spouted bed of hot air at a rate of 4 m.sup.3 /min. After the temperature was elevated to a given value, a given resin solution was fed at a rate of 300 ml/hr to carry out the coating treatment. The resin used in this Example is Stylon No. 679 manufactured by Asahi Dow which is a polystyrene resin having a relatively low molecular weight. As for solvent, toluene was used, and 5%, 2% and 1% solutions of resin were prepared therefrom and used. Further, coating treatment was carried out at granule temperatures of 50.degree., 60.degree., 70.degree. and 80.degree. C, and in either

cases, agglomeration of granules occurred. Within one minute after the respective resin solutions were fed, fluidization stopped and no capsule could be obtained.

Brief Summary Text (180):

Using the same apparatus as in Example 8, 5 kg of granular <u>fertilizer</u> used in Example 2 was spouted by the stream of hot air at a flow rate of 4 m.sup.3 /min, and after the granule temperature reached 60.degree. C, solutions of polymers shown in Table 16 were fed at a rate of 500 g/min, for 10 minutes to give products. Under these conditions, the temperature of ejected air for drying was maintained at 90.degree. C, and the granule temperature was maintained at 60.degree. C. The nitrogen dissolving-out percent and extent of agglomeration of granules are shown in Table 16.

Brief Summary Text (193):

6. Size of fertilizer granule: 1-10 mm, preferably 2-6 mm

Brief Summary Paragraph Table (5):	
Table 5	Granule Velocity tempera- After After
of air ture 24 hours one month Appearance	<u> </u>
50.degree. C 1.0% 3.8% Lustrous, trans- paren	nt and uni- form capsule, 60.degree. C
1.2% 4.7% capsule peeled 15 off from m/sec gr	canular 70.degree. C 1.8% 4.9%
fertilizer exhibits a tough film 80.degree. C	2 2.4% 10.3% state 50.degree. C 0.3%
0.9% 60.degree. C 0.3% 1.1% 30 " m/sec 70.deg	ree. C 0.4% 2.9% 80.degree. C 1.9% 4.9%

CLAIMS:

- 1. A method for producing slow-releasing granular <u>fertilizer</u> fully coated with water-resisting, transparent, lustrous layer of resin in such a manner that the coated granules do not stick together, which comprises spraying a solution having a viscosity of 40 C.P. or less at close to its boiling point of a thermoplastic resin in a hydrocarbon or chlorinated hydrocarbon solvent, upon <u>fertilizer</u> granules, and drying said <u>fertilizer</u> granules upon being sprayed with said solution, continuously and immediately on the spout with hot, dry air at an air speed of at least 15m/sec., said solution having the property capable of forming a solution while it is hot but turning into jelly-like gel when it is cooled and said granules being at a temperature of 40.degree.-90.degree. C during the drying and spraying steps.
- 10. A method for producing slow-releasing granular <u>fertilizer</u> fully coated with water-resisting transparent, lustrous layer of resin in such a manner that the coated granules do not stick together, which comprises spraying a solution having a viscosity of 40 C.P. or lower at close to its boiling point of a thermoplastic resin and an additive thereto, in a hydrocarbon or chlorinated hydrocarbon solvent, upon <u>fertilizer</u> granules, and drying said <u>fertilizer</u> granules upon being sprayed with said solution, continuously and immediately on the spot with hot, dry air at an air speed of at least 15m/sec., said solution having the property capable of forming a solution while it is hot but turning into jelly-like gel when it is cooled, said granules being at a temperature of 40.degree.-90.degree. C during the drying and spraying steps.

Generate Collection Print

L12: Entry 9 of 10 File: DWPI Feb 25, 1993

DERWENT-ACC-NO: 1993-067839

DERWENT-WEEK: 200039

COPYRIGHT 2003 DERWENT INFORMATION LTD

TITLE: Fertiliser granules coated with several distinct layers of polyurethane formed by hardening poly:isocyanate-poly:ol coating mixt., provide sustained,
diffusion controlled nutrient release

INVENTOR: BURGER, H; JASCHKOWITZ, M; KLOTH, B; KOHL, W; WEGENER, H; WEHR, P; BUERGER, H

PATENT-ASSIGNEE: AGLUKON SPEZIALDUNGER GMBH (AGLUN), AGLUKON SPEZIALDUENGER GMBH (AGLUN), AGLUCON SPEZIALDUNGER GMBH (AGLUN)

PRIORITY-DATA: 1991DE-4127459 (August 20, 1991)

PATENT-FAMILY:

PU	B-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
DΕ	4127459 A1	February 25, 1993		011	C05G003/00
HU	218166 B	June 28, 2000		000	C05G003/00
WO	9304017 A1	March 4, 1993	G	022	C05G003/00
AU	9224399 A	March 16, 1993		000	C05G003/00
DE	4127459 C2	July 29, 1993		011	C05G003/00
ZA	9206284 A	July 28, 1993		019	C05G000/00
PT	100787 A	September 30, 1993		000	C05G005/00
NO	9400386 A	February 7, 1994		000	C05G003/00
FI	9400776 A	February 18, 1994		000	C05G000/00
EP	599927 A1	June 8, 1994	G	000	C05G003/00
CZ	9400367 A3	July 13, 1994		000	C05G003/00
AU	652663 B	September 1, 1994		000	C05G005/00
JP	07500560 W	January 19, 1995	•	000	C05G003/00
NZ	244013 A	February 24, 1995		000	C05G005/00
HU	70289 T	September 28, 1995		000	C05G003/00
ΙL	102868 A	October 31, 1995		000	C05G003/00
ΕP	599927 B1	June 11, 1997	G	011	C05G003/00
DE	59208613 G	July 17, 1997		000	C05G003/00
ES	2104939 T3	October 16, 1997		000	C05G003/00
CZ	282958 B6	November 12, 1997		000	C05G003/00
RU	2091357 C1	September 27, 1997		800	C05G003/00
NO	303908 B1	September 21, 1998		000	C05G003/00

DESIGNATED-STATES: AU CA CS FI HU JP NO PL RU US AT BE CH DE DK ES FR GB GR IE IT LU MC NL SE AT BE CH DE DK ES FR GB GR IE IT LI LU MC NL SE AT BE CH DE DK ES FR GB GR IE IT LI LU NL SE

CITED-DOCUMENTS:DE 3544451; US 3264089 ; US 4711659

APPLICATION-DATA	:		
PUB-NO	APPL-DATE	APPL-NO	DESCRIPTOR
DE 4127459A1	August 20, 1991	1991DE-4127459	
HU 218166B	August 18, 1992	1992WO-DE00697	
HU 218166B	August 18, 1992	1994HU-0000479	
HU 218166B		HU 70289	Previous Publ.
HU 218166B		WO 9304017	Based on
WO 9304017A1	August 18, 1992	1992WO-DE00697	
AU 9224399A	August 18, 1992	1992AU-0024399	
AU 9224399A		WO 9304017	Based on
DE 4127459C2	August 20, 1991	1991DE-4127459	
ZA 9206284A	August 20, 1992	1992ZA-0006284	
PT 100787A	August 19, 1992	1992PT-0100787	
NO 9400386A	August 18, 1992	1992WO-DE00697	
NO 9400386A	February 7, 1994	1994NO-0000386	
FI 9400776A	August 18, 1992	1992WO-DE00697	
FI 9400776A	February 18, 1994	1994FI-0000776	
EP 599927A1	August 18, 1992	1992EP-0917606	
EP 599927A1	August 18, 1992	1992WO-DE00697	
EP 599927A1	•	WO 9304017	Based on
CZ 9400367A3	August 18, 1992	1994CZ-0000367	
AU 652663B	August 18, 1992	1992AU-0024399	
AU 652663B		AU 9224399	Previous Publ.
AU 652663B		WO 9304017	Based on
JP 07500560W	August 18, 1992	1992WO-DE00697	
JP 07500560W	August 18, 1992	1993JP-0504031	
JP 07500560W		WO 9304017	Based on
NZ 244013A	August 19, 1992	1992NZ-0244013	
HU 70289T	August 18, 1992	1992WO-DE00697	
HU 70289T	August 18, 1992	1994HU-0000479	
HU 70289T		WO 9304017	Based on
IL 102868A	August 19, 1992	1992IL-0102868	
EP 599927B1	August 18, 1992	1992EP-0917606	
EP 599927B1	August 18, 1992	1992WO-DE00697	
EP 599927B1		WO 9304017	Based on
DE 59208613G	August 18, 1992	1992DE-0508613	
DE 59208613G	August 18, 1992	1992EP-0917606	
DE 59208613G	August 18, 1992	1992WO-DE00697	
DE 59208613G		EP 599927	Based on
DE 59208613G		WO 9304017	Based on
ES 2104939T3	August 18, 1992	1992EP-0917606	•
ES 2104939T3		EP 599927	Based on
CZ 282958B6	August 18, 1992	1992WO-DE00697	
CZ 282958B6	August 18, 1992	1994CZ-0000367	
CZ 282958B6		CZ 9400367	Previous Publ.
CZ 282958B6		WO 9304017	Based on
RU 2091357C1	August 18, 1992	1992WO-DE00697	
RU 2091357C1	August 18, 1992	1994RU-0015604	
NO 303908B1	August 18, 1992	1992WO-DE00697	
NO 303908B1	February 7, 1994	1994NO-0000386	
NO 303908B1		NO 9400386	Previous Publ.

599927 B1 INT-CL (IPC): B29B 9/16; C05G 0/00; C05G 3/00; C05G 3/10; C05G 5/00

ABSTRACTED-PUB-NO: DE 4127459A BASIC-ABSTRACT:

Prepn. of membrane-coated <u>fertiliser</u> granules comprises applying in layers, a mixt. of polyisocyanate (I) and polyol (II). The new feature is that the coating mass is applied to form layers each 10-30 (pref. 15-20) microns thick.

Each portion of coating mass is individually hardened with a liq., atomised amine, and after each hardening step the amine concn. is reduced to an inactive level before applying the next layer.

USE/ADVANTAGE - The granules preferentially release N in the early stages, and K later. They can be prepd. economically and on a large scale, with high homogeneity in each layer; are physically stable, resistant to frost and provide sustained release of nutrients in accordance with the laws of diffusion. The granules can be used alone or combined with other fertiliser

ABSTRACTED-PUB-NO: DE 4127459C EQUIVALENT-ABSTRACTS:

<u>Fertiliser</u> granules with a membrane coating are produced by forming layers of polyisocyanate and polyol, with each layer hardened separately with a mist of liq. amine. The individual layers are 10-30 microns thick. Between each hardening process, the amine concn. is reduced using a non-catalytic water gauge, before the next layer is applied. Pref. the mixt. is supplied at a temp. of 25-50 deg.C.

USE/ADVANTAGE - The delayed release <u>fertiliser</u> can be designed to release different components at different times, e.g. K may be released more slowly or later than N.

EP 599927B

A process for the manufacture of membrane-covered <u>fertiliser</u> granules by applying layers of a mixture of polyisocyanate and polyol, the layers each being cured separately with amine nebulized in liquid form, wherein the mixture is applied in each case in amounts such that the individual layers have a layer thickness of from 10 to 30 mm and, after the curing of each layer, the amine concentration is reduced to a level which is not catalytically active before the next layer is applied.

CHOSEN-DRAWING: Dwg.0/8 Dwg.0/8 Dwg.0/8

DERWENT-CLASS: A97 C04

CPI-CODES: A05-G01E1; A11-B05C; A12-B; A12-W04B; C04-C03D; C05-B02A4; C10-E04C; C12-M10; C12-M10A; C12-M1D; C12-N09; C12-N10;

End of Result Set

Generate Collection Print

L12: Entry 10 of 10

File: DWPI

DERWENT-ACC-NO: 1968-63337P

DERWENT-WEEK: 196800

COPYRIGHT 2003 DERWENT INFORMATION LTD

TITLE: Slow release fertiliser coated with resin comprising: - (a) a core of granular

fertiliser (at least one soluble nutrient contg. N, P or K); (b) a num

PATENT-ASSIGNEE: ARCHER-DANIELS MIDLAND CORP (ARCH)

PRIORITY-DATA: 1965US-0496135 (October 14, 1965), 1961US-0132383 (August 18, 1961)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES

MAIN-IPC

US 3259482 A

000

ABSTRACTED-PUB-NO: US 3259482A

BASIC-ABSTRACT:

A coated <u>fertilizer</u> comprises (a) a core of <u>granular fertilizer</u> containing at least one water soluble plant nutrient which is a compound of N, P, or K; (b) <u>several cured coatings</u> of an epoxy/polyester resin system surrounding the core, each coating being 0.1 - 10% by weight of the core, the coating weight being such as to <u>delay the release</u> of the plant nutrient. The coatings are the reaction product of (1) an epoxy component consisting of at least 80% of epoxidized fatty compound having 8-26 C atoms in its fatty radicals and an internal oxirane value of 3-10% and (2) a polyester curing agent which is the esterification reaction product of polyhydric alcohol and polycarboxylic acid.

ABSTRACTED-PUB-NO: US 3259482A EQUIVALENT-ABSTRACTS:

DERWENT-CLASS: A00 C00

DERWENT-CLASS: AUU CUU

CPI-CODES: A05-A01; A08-D; A12-B; A12-W04; C04-C03; C05-A01A; C05-B02A; C05-C01; C05-C02; C10-A13B; C12-M10; C12-N09; C12-N10;

Generate Collection

Print

Search Results - Record(s) 1 through 1 of 1 returned.

☐ 1. Document ID: US 4019890 A

L18: Entry 1 of 1

File: USPT

Apr 26, 1977

US-PAT-NO: 4019890

DOCUMENT-IDENTIFIER: US 4019890 A

TITLE: Method for producing coated fertilizer

DATE-ISSUED: April 26, 1977

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP	CODE	COUNTRY
Fujita; Toshio	Fujishi			•	JA
Takahashi; Chigo	Fujishi				JA
Ohshima; Masanari	Fujishi				JA
Ushioda; Tsunezo	Tokyoto				JA
Shimizu; Hirozo	Fujishi				JA

US-CL-CURRENT: 71/64.11; 427/212, 71/35

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Drawu D	esc I	mage								
				~~,	***************************************					
					Generate C	:ollecti	ion	Print		
				ļ	Concrate C	, On COL				
							7			
			7	Cerms				Docu	ments	
111 same L17						1		•	1	
	HIII D	anic Li	,				11			11

Display Format: - Change Format

Previous Page Next Page

End of Result Set

Generate Collection Print

L18: Entry 1 of 1

File: USPT

Apr 26, 1977

DOCUMENT-IDENTIFIER: US 4019890 A

TITLE: Method for producing coated fertilizer

Brief Summary Text (32):

The valuableness of the present invention is in the capability of controlling the dissolving rate of product. In the conventional methods such as a multiple coating method which uses a thermosetting resin or sulfur coating method, the release of fertilizer component has been controlled by providing pinholes in the coating of granules formed during preparation or by decomposition of sulfur by soil microorganisms. However, in the method of the present invention, the control of dissolving is carried out by providing practically insoluble granular fertilizers completely coated with very thin film in the first step and by adding a certain additive e.g. a hydrophilic substance in the coating films to give an arbitrary dissolving speed in the second place.

Generate Collection

Print

Search Results - Record(s) 1 through 10 of 24 returned.

☐ 1. Document ID: US 6338847 B1

L19: Entry 1 of 24

File: USPT

Jan 15, 2002

US-PAT-NO: 6338847

DOCUMENT-IDENTIFIER: US 6338847 B1

TITLE: Compositions and methods to disinfect contact lenses

DATE-ISSUED: January 15, 2002

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Thomas; Larry K. Fullerton CA

US-CL-CURRENT: 424/94.2; 422/30, 424/94.4, 424/94.6, 424/94.61, 424/94.62,

424/94.63, 424/94.64, 424/94.65, 424/94.66, 424/94.67

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

KMAC

☐ 2. Document ID: US 6165954 A

L19: Entry 2 of 24

File: USPT

Dec 26, 2000

KMMC

US-PAT-NO: 6165954

DOCUMENT-IDENTIFIER: US 6165954 A

TITLE: Enzyme compositions and methods for contact lens cleaning

DATE-ISSUED: December 26, 2000

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Huth; Stanlev W. Newport Beach CA

US-CL-CURRENT: <u>510/114</u>; <u>510/112</u>, <u>510/383</u>, <u>510/384</u>, <u>510/391</u>, <u>510/392</u>, <u>510/530</u>

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Descripting Control of the Control

☑ 3. Document ID: US 6046177 A

L19: Entry 3 of 24 File: USPT Apr 4, 2000

US-PAT-NO: 6046177

DOCUMENT-IDENTIFIER: US 6046177 A

TITLE: Sulfoalkyl ether cyclodextrin based controlled release solid pharmaceutical

formulations

DATE-ISSUED: April 4, 2000

INVENTOR - INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Stella; Valentino J.

Rajewski; Roger A.

Lawrence

KS
Rao; Venkatramana M.

Lawrence

KS
McGinity; James W.

Mosher; Gerold L.

Lawrence

KS
Austin

TX
Mosher; Gerold L.

Kansas City

MO

US-CL-CURRENT: 514/58; 514/778, 514/964, 514/965, 536/103

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC |
Draw, Desc | Image |

☐ 4. Document ID: US 5849060 A

L19: Entry 4 of 24

File: USPT Dec 15, 1998

US-PAT-NO: 5849060

DOCUMENT-IDENTIFIER: US 5849060 A

TITLE: Controlled release fertilizer and preparations thereof

DATE-ISSUED: December 15, 1998

INVENTOR-INFORMATION:

NAME STATE ZIP CODE COUNTRY CITY Diping; Li CN Zhengzhou CN Haobin; Wang Zhengzhou Xiucheng; Xu CN Zhengzhou Cuihong; Hou Zhengzhou CN

US-CL-CURRENT: 71/64.07; 71/28, 71/29, 71/30, 71/31, 71/32, 71/33, 71/34, 71/35, 71/36, 71/64.11

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

5. Document ID: US 5840250 A

L19: Entry 5 of 24

File: USPT

Nov 24, 1998

US-PAT-NO: 5840250

DOCUMENT-IDENTIFIER: US 5840250 A

TITLE: Compositions and methods for disinfecting a contact lens and detecting the presence of an oxidative disinfectant

DATE-ISSUED: November 24, 1998

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Park; John Y.

Santa Ana

CA

Peng; Lin

Tustin

CA

Dziabo; Anthony J.

Lake Forest

CA

US-CL-CURRENT: 422/28; 134/42, 422/29, 422/30, 510/100, 510/112, 510/113, 510/115, 510/298, 510/373

Full Title Citation Front Review Classification Date Reference Sequences Attachments Draw, Desc | Image

☐ 6. Document ID: US 5783532 A

L19: Entry 6 of 24

File: USPT

Jul 21, 1998

US-PAT-NO: 5783532

DOCUMENT-IDENTIFIER: US 5783532 A

TITLE: Enzyme compositions and methods for contact lens cleaning

DATE-ISSUED: July 21, 1998

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Huth; Stanley W.

Newport Beach

CA

US-CL-CURRENT: 510/114; 435/184, 510/392, 510/393, 510/530, 514/839

Title Citation Front Review Classification Date Reference Sequences Attachments Draw, Desc Image

7. Document ID: US 5766931 A

L19: Entry 7 of 24

File: USPT

Jun 16, 1998

US-PAT-NO: 5766931

DOCUMENT-IDENTIFIER: US 5766931 A

TITLE: Composition and methods for destroying hydrogen peroxide

DATE-ISSUED: June 16, 1998

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Cook; James N. Mission Viejo CA Worsley; John L. Irvine CA

US-CL-CURRENT: <u>435/264</u>; <u>422/30</u>, <u>424/468</u>, <u>424/94.4</u>, <u>435/192</u>, <u>435/917</u>

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

☐ 8. Document ID: US 5746972 A

L19: Entry 8 of 24

File: USPT

May 5, 1998

US-PAT-NO: 5746972

DOCUMENT-IDENTIFIER: US 5746972 A

TITLE: Compositions and methods for disinfecting and cleaning contact lenses

DATE-ISSUED: May 5, 1998

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Park; John Y. Santa Ana CA
Thomas; Larry K. Irvine CA
Peng; Lin Sunnyvale CA
Cafaro; Daniel P. Walnut Creek CA

US-CL-CURRENT: 422/30; 422/29, 435/192, 435/264, 514/840

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

☐ 9. Document ID: US 5746838 A

L19: Entry 9 of 24

File: USPT

May 5, 1998

US-PAT-NO: 5746838

DOCUMENT-IDENTIFIER: US 5746838 A

TITLE: Enzyme compositions and methods for contact lens cleaning

DATE-ISSUED: May 5, 1998

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Huth; Stanley W. Newport Beach CA

US-CL-CURRENT: <u>134/27</u>; <u>134/28</u>, <u>134/901</u>, <u>435/184</u>, <u>514/839</u>, <u>514/840</u>

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC Draw, Description

☐ 10. Document ID: US 5719110 A

L19: Entry 10 of 24

File: USPT

Feb 17, 1998

US-PAT-NO: 5719110

DOCUMENT-IDENTIFIER: US 5719110 A

ZIP CODE

TITLE: Contact lens care compositions with inositol phosphate components

DATE-ISSUED: February 17, 1998

INVENTOR-INFORMATION:

NAME CITY

STATE

COUNTRY

Cook; James N.

Mission Viejo

CA

US-CL-CURRENT: 510/112; 134/42, 435/264, 510/113, 510/114, 510/382, 510/383, 510/462, 510/463, 510/468, 510/469

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWC
Draw, D	esc Ir	mage								
					Generate C	Collecti	on	Print		
				E		***************************************	F	***************************************		
				T					\	
	Terms							L	ocuments	
	(111 and L17) not 118									24
	11	— - · .	,				j.	i .		11

Display Format: - Change Format

Previous Page Next Page

Generate Collection

Print

Search Results - Record(s) 11 through 20 of 24 returned.

☐ 11. Document ID: US 5703024 A

L19: Entry 11 of 24

File: USPT

Dec 30, 1997

US-PAT-NO: 5703024

DOCUMENT-IDENTIFIER: US 5703024 A

TITLE: Compositions and methods for disinfecting a contact lens and detecting the

presence of an oxidative disinfectant

DATE-ISSUED: December 30, 1997

INVENTOR-INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

Park; John Y.

Santa Ana

CA

Peng; Lin

Tustin

CA CA

Dziabo; Anthony J.

Lake Forest

US-CL-CURRENT: 510/100; 252/408.1, 510/112, 510/113, 510/115, 510/373

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw, D	esc li	mage							

KMIC

☐ 12. Document ID: US 5648074 A

L19: Entry 12 of 24

File: USPT

Jul 15, 1997

US-PAT-NO: 5648074

DOCUMENT-IDENTIFIER: US 5648074 A

TITLE: Compositions and methods for disinfecting contact lenses and reducing

proteinaceous deposit formation

DATE-ISSUED: July 15, 1997

INVENTOR-INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

Park; John Y.

Santa Ana

CA

Peng; Lin

Tustin

CA

Dziabo; Anthony J.

Lake Forest

CA

US-CL-CURRENT: <u>424/94.2</u>; <u>424/661</u>, <u>424/94.1</u>, <u>424/94.63</u>, <u>435/264</u>, <u>514/54</u>, <u>514/56</u>, <u>514/57</u>

Full Title Citation Front Review Classification Date Reference Sequences Attachments

KAMC

☐ 13. Document ID: US 5630884 A

L19: Entry 13 of 24

File: USPT

May 20, 1997

US-PAT-NO: 5630884

DOCUMENT-IDENTIFIER: US 5630884 A

TITLE: Methods for contact lens cleaning

DATE-ISSUED: May 20, 1997

INVENTOR - INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

Huth; Stanley W.

Newport Beach

CA

US-CL-CURRENT: 134/27; 134/901, 435/184, 514/839

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

KWIC

☐ 14. Document ID: US 5549891 A

L19: Entry 14 of 24

File: USPT

Aug 27, 1996

US-PAT-NO: 5549891

DOCUMENT-IDENTIFIER: US 5549891 A

TITLE: Method for disinfecting contact lens with catalase compositions

DATE-ISSUED: August 27, 1996

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Sulc; Jiri S. Krcova; Zuzana

La Jolla

Newport Beach

CA CA

US-CL-CURRENT: $\frac{424}{94.4}$; $\frac{424}{458}$, $\frac{424}{462}$, $\frac{424}{464}$, $\frac{424}{468}$, $\frac{424}{469}$, $\frac{424}{469}$, $\frac{424}{471}$, $\frac{424}{94.2}$, $\frac{424}{94.3}$

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

KWC

☐ 15. Document ID: US 5521091 A

L19: Entry 15 of 24

File: USPT

May 28, 1996

US-PAT-NO: 5521091

DOCUMENT-IDENTIFIER: US 5521091 A

TITLE: Compositions and method for destroying hydrogen peroxide on contact lens

DATE-ISSUED: May 28, 1996

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Cook; James N. M

Mission Viejo

CA

CA

Worsley; John L. Irvine

US-CL-CURRENT: 435/264; 422/30, 424/468, 424/94.4, 435/192

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

16. Document ID: US 5510119 A

L19: Entry 16 of 24

File: USPT

Apr 23, 1996

US-PAT-NO: 5510119

DOCUMENT-IDENTIFIER: US 5510119 A

TITLE: Controlled release therapeutic system for liquid pharmaceutical formulations

DATE-ISSUED: April 23, 1996

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Santus; Giancarlo Milan IT Golzi; Roberto Cremona IT

US-CL-CURRENT: 424/490; 424/493, 424/494, 424/495, 424/498

Full Title Citation Front Review Classification Date Reference Sequences Attachments

NMC |
Draw, Desc | Image |

☐ 17. Document ID: US 5362647 A

L19: Entry 17 of 24

File: USPT

Nov 8, 1994

US-PAT-NO: 5362647

DOCUMENT-IDENTIFIER: US 5362647 A

TITLE: Compositions and methods for destroying hydrogen peroxide

DATE-ISSUED: November 8, 1994

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Cook; James N. Mission Viejo CA Worsley; John L. Irvine CA

US-CL-CURRENT: $\frac{435}{264}$; $\frac{422}{30}$, $\frac{424}{406}$, $\frac{424}{417}$, $\frac{510}{114}$, $\frac{510}{392}$, $\frac{510}{446}$

Full Title Citation Front Review Classification Date Reference Sequences Attachments

KORAC

☐ 18. Document ID: US 5338480 A

L19: Entry 18 of 24

File: USPT

Aug 16, 1994

US-PAT-NO: 5338480

DOCUMENT-IDENTIFIER: US 5338480 A

TITLE: Compositions and methods to clean contact lenses

DATE-ISSUED: August 16, 1994

INVENTOR - INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Dziabo; Anthony J. El Toro CA
Karageozian; Hampar Laguna Hills CA
Ripley; Paul S. Irvine CA
Lam; Sam W. Laguna Niguel CA
Espiritu; J. Abraham Oceanside CA

US-CL-CURRENT: 252/187.21; 252/187.23, 252/187.25, 252/187.26, 252/187.27, 424/661, 424/662, 435/264

Full Title Citation Front Review Classification Date Reference Sequences Attachments KWC Draw, Description

☐ 19. Document ID: US 5336434 A

L19: Entry 19 of 24

File: USPT

Aug 9, 1994

US-PAT-NO: 5336434

DOCUMENT-IDENTIFIER: US 5336434 A

TITLE: Methods, compositions and apparatus to disinfect lenses

DATE-ISSUED: August 9, 1994

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Park; John Y. Santa Ana CA Ripley; Paul S. Irvine CA Dziabo; Anthony J. Lake Forest CA

US-CL-CURRENT: 252/187.21; 424/661, 435/264

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC

Draw Desc Image

☐ 20. Document ID: US 5324447 A

L19: Entry 20 of 24 File: USPT Jun 28, 1994

US-PAT-NO: 5324447

DOCUMENT-IDENTIFIER: US 5324447 A

TITLE: Method and activator compositions to disinfect lenses

DATE-ISSUED: June 28, 1994

INVENTOR-INFORMATION:

NAME ·

CITY

STATE ZIP CODE

COUNTRY

Lam; Sam W.

Laguna Niguel

CA

Ripley; Paul S.

Irvine

CA

Espiritu; J. Abraham M.

Oceanside

CA

US-CL-CURRENT: $\underline{252}/\underline{187.21}$; $\underline{252}/\underline{187.23}$, $\underline{252}/\underline{187.25}$, $\underline{252}/\underline{187.26}$, $\underline{252}/\underline{187.26}$, $\underline{252}/\underline{187.27}$, $\underline{424}/\underline{661}$, $\underline{424}/\underline{662}$, $\underline{435}/\underline{264}$

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	К	MC
Drawi D	esc Ir	mage								` -	
					Generate C	\allaak		Delea			
				L	Generale	onecu	on]	Print			
				<u> </u>	Generale	onecu	on II	Print			
				L Ter		Jonecu	on IL				
		and L17		Ter		Jonecu	On] [Ocuments		

Display Format: - Change Format

Previous Page Next Page

Generate Collection

Print

Search Results - Record(s) 21 through 24 of 24 returned.

☑ 21. Document ID: US 5306440 A

L19: Entry 21 of 24

File: USPT

Apr 26, 1994

COUNTRY

US-PAT-NO: 5306440

DOCUMENT-IDENTIFIER: US 5306440 A

TITLE: Methods for generating chlorine dioxide and compositions for disinfecting

DATE-ISSUED: April 26, 1994

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE

Ripley; Paul S. Irvine CA
Dziabo; Anthony J. El Toro CA

Ringo; James P. Norman OK

US-CL-CURRENT: <u>252/186.33</u>; 252/186.36, 252/187.21, 252/187.23, 424/661

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

KWIC

☐ 22. Document ID: US 5296236 A

L19: Entry 22 of 24

File: USPT

Mar 22, 1994

US-PAT-NO: 5296236

DOCUMENT-IDENTIFIER: US 5296236 A

TITLE: Controlled release therapeutic system for a liquid pharmaceutical

formulations

DATE-ISSUED: March 22, 1994

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Santus; Giancarlo Milan IT
Golzi; Roberto Cremona IT

US-CL-CURRENT: 424/484; 424/440

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Drawl Desc | Image |

KOMO

☐ 23. Document ID: US 5246662 A

L19: Entry 23 of 24

File: USPT

Sep 21, 1993

US-PAT-NO: 5246662

DOCUMENT-IDENTIFIER: US 5246662 A

TITLE: Methods for generating chlorine dioxide and compositions for disinfecting

DATE-ISSUED: September 21, 1993

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Ripley; Paul S. Irvine
Dziabo; Anthony J. El Toro

El Toro CA

CA

Ringo; James P. Norman OK

US-CL-CURRENT: 422/29; 252/187.21, 252/187.23, 422/37

Full Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Draw, Desc | Image |

☐ 24. Document ID: US 5078908 A

L19: Entry 24 of 24

File: USPT

Jan 7, 1992

US-PAT-NO: 5078908

DOCUMENT-IDENTIFIER: US 5078908 A

TITLE: Methods for generating chlorine dioxide and compositions for disinfecting

DATE-ISSUED: January 7, 1992

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Ripley; Paul S. Irvine CA
Dziabo; Anthony J. El Toro CA
Ringo; James P. Norman OK

US-CL-CURRENT: 252/187.21; 252/186.33, 252/186.36, 252/187.23, 424/661

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC |
Draw, Desc | Image |

Generate Collection Print

Terms	Documents
(111 and L17) not 118	24

Generate Collection Print

L19: Entry 3 of 24

File: USPT

Apr 4, 2000

DOCUMENT-IDENTIFIER: US 6046177 A

TITLE: Sulfoalkyl ether cyclodextrin based controlled release solid pharmaceutical

formulations

Detailed Description Text (106):

As used herein, the term "preservative" is intended to mean a compound used to prevent the growth of microorganisms. Such compounds include, by way of example and without limitation, benzalkonium chloride, benzethonium chloride, benzyl alcohol, cetylpyridinium chloride, chlorobutanol, phenol, phenylethyl alcohol, phenylmercuric nitrate and thimerosal and the like.

CLAIMS:

- 28. The <u>multi-layered controlled release</u> solid pharmaceutical formulation of claim 23 wherein said formulation is a tablet, minitablet, granule, pellet or micropellet.
- 33. The <u>multi-layered</u> combined rapid and <u>controlled release</u> solid pharmaceutical formulation of claim 29 wherein said formulation is a tablet, minitablet, <u>granule</u>, pellet or micropellet.
- 38. The <u>multi-layered</u> combined rapid and <u>controlled release</u> solid pharmaceutical formulation of claim 34 wherein said formulation is a tablet, minitablet, <u>granule</u>, pellet or micropellet.

Generate Collection Print

L19: Entry 16 of 24

File: USPT

Apr 23, 1996

DOCUMENT-IDENTIFIER: US 5510119 A

TITLE: Controlled release therapeutic system for liquid pharmaceutical formulations

Brief Summary Text (40):

In addition to the amount of coated microgranules containing the dose of controlled release active ingredient, the elements constituting the vehicle for form (a) are: (1)--a dose of active principle in solution, forming a readily absorbable fraction; (2) suspending and structural agents such as cellulose esters, microcrystalline cellulose, alginic acid derivatives, polyvinyl pyrroidone derivatives; (3) sugars, such as sucrose and sorbitol; (4) buffers such as citric acid and sodium citrate, glycine and hydrochloric acid, sodium and potassium phosphates; (5) preservatives and bacteriostatic agents such as p-hydroxybenzoic acid esters; (6) aromatizing and sweetening agents such as saccharine and others; and (7) water or mixtures of water and solvents such as glycols, alcohols, glycerin.

Detailed Description Text (31):

The advantage of the complete <u>multiple coating</u> (E) consists in keeping reduced <u>granule</u> dimensions while maintaining the efficacy in <u>delaying the release</u> of the active ingredient, notwithstanding the high surface area of the particles.

Generate Collection Print

L19: Entry 21 of 24

File: USPT

Apr 26, 1994

DOCUMENT-IDENTIFIER: US 5306440 A

TITLE: Methods for generating chlorine dioxide and compositions for disinfecting

Brief Summary Text (48):

Although <u>multi-layered</u> (including core and coating layering) tablets or pills are preferred, the <u>delayed release</u> form of the present components can be present in any other suitable item or items, such as masses of powders, <u>granules</u> and the like. Delayed release technology is well known in the art as exemplified by the text Controlled Drug Delivery, 2nd Ed., Joseph R. Robinson and Vincent H. L. Lee, Eds., Marcel Dekker, Inc., New York, 1987.

Detailed Description Text (45):

A solution containing deionized water, 0.85% (w/v) of sodium chloride, 0.10% (w/v) of boric acid, and 50 pp. w/v of the stabilized chlorine dioxide product identified in Example 1 was prepared. One portion of this solution was buffered to a pH 7.9, while the other portion was buffered to a pH of 6.8. Varying amounts of tartaric acid was added to different aliquots of each of these portions. The aliquots were then tested, following the standard procedure, to determine the D-value with respect to various microorganisms. The D-value is defined as the length of time required to reduce the microbial burden or load by one log unit.

Detailed Description Paragraph Table (9):

TABLE 9

pH = 6.8 Tartaric Acid, ppm. 30 40 50 60 70 Free Chlorine 10.74 17.08 37.94 25.38 32.47 Dioxide, ppm Microorganism

Extrapolated D-value at 23.degree. C., min. S. marcescens <0.84 <0.84 <0.84 <0.84 <0.84 <0.84 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.85 <0.

WEST

Generate Collection

Print

Search Results - Record(s) 1 through 9 of 9 returned.

☐ 1. Document ID: US 20020098982 A1

L21: Entry 1 of 9

File: PGPB

Jul 25, 2002

PGPUB-DOCUMENT-NUMBER: 20020098982

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020098982 A1

TITLE: Production and use of biosolid granules

PUBLICATION-DATE: July 25, 2002

INVENTOR-INFORMATION:

NAME

CITY

STATE

COUNTRY

RULE-47

Burnham, Jeffrey C.

Naples

FL

US

US-CL-CURRENT: 504/359; 504/117, 504/367, 71/64.02, 71/64.11

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw. Desc | Image |

KOMO:

2. Document ID: US 6325934 B1

L21: Entry 2 of 9

File: USPT

Dec 4, 2001

US-PAT-NO: 6325934

DOCUMENT-IDENTIFIER: US 6325934 B1

TITLE: Enzyme and bacterial combination in a slowly dissolvable matrix for septic

tanks, grease traps and waste treatment

DATE-ISSUED: December 4, 2001

 ${\tt INVENTOR-INFORMATION:}$

NAME CITY STATE ZIP CODE

Tobey, Jr.; James Francis Salem VA 24153 Stapleton, Jr.; Raymond D. Staunton VA 24401

US-CL-CURRENT: 210/606; 210/611, 210/616, 210/631, 210/632, 435/177, 435/180,

435/182

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

KMC

COUNTRY

☑ 3. Document ID: US 5939086 A

L21: Entry 3 of 9

File: USPT

Aug 17, 1999

US-PAT-NO: 5939086

DOCUMENT-IDENTIFIER: US 5939086 A

TITLE: Compositions and methods for reducing the amount of contaminants in aquatic

and terrestrial environments

DATE-ISSUED: August 17, 1999

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Levy; Richard

Fort Myers

FL

US-CL-CURRENT: 424/405; 424/404, 424/406, 424/407, 424/408, 424/409, 424/417, 424/418, 424/419, 424/78.18, 504/360, 525/54.31, 525/54.32



KWIC

☐ 4. Document ID: US 5885602 A

L21: Entry 4 of 9

File: USPT

Mar 23, 1999

US-PAT-NO: 5885602

DOCUMENT-IDENTIFIER: US 5885602 A

TITLE: Process for the persistence control of chemicals released into the

environment

DATE-ISSUED: March 23, 1999

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Levy; Richard

Fort Myers

FL

US-CL-CURRENT: $\underline{424}/\underline{405}$; $\underline{424}/\underline{404}$, $\underline{424}/\underline{406}$, $\underline{424}/\underline{407}$, $\underline{424}/\underline{408}$, $\underline{424}/\underline{409}$, $\underline{424}/\underline{417}$, 424/78.18, 523/124, 524/10, 524/9

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KOMO
Drawt D	eso li	mage								

☑ 5. Document ID: US 5863789 A

L21: Entry 5 of 9

File: USPT

Jan 26, 1999

US-PAT-NO: 5863789

DOCUMENT-IDENTIFIER: US 5863789 A

TITLE: Microorganism-holding carrier and method for remediation of soil employing

the carrier

DATE-ISSUED: January 26, 1999

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY Komatsu; Toshiyuki Hiratsuka JΡ Sakuranaga; Masanori Atsugi JP Nomoto; Tsuyoshi Atsuqi JΡ Kozaki; Shinya JΡ Tokyo Imamura; Takeshi Chigasaki JP

US-CL-CURRENT: $\frac{435}{262}$; $\frac{435}{178}$, $\frac{435}{179}$, $\frac{435}{180}$, $\frac{435}{244}$, $\frac{435}{245}$, $\frac{435}{262.5}$, $\frac{435}{110}$

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC Draw, Desc Image

☐ 6. Document ID: US 5679364 A

L21: Entry 6 of 9

File: USPT

Oct 21, 1997

US-PAT-NO: 5679364

DOCUMENT-IDENTIFIER: US 5679364 A

TITLE: Compositions and methods for reducing the amount of contaminants in aquatic

and terrestrial environments

DATE-ISSUED: October 21, 1997

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Levy; Richard Fort Myers FL

US-CL-CURRENT: 424/405; 424/406, 424/409, 424/417, 424/78.18

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

☐ 7. Document ID: US 5476992 A

L21: Entry 7 of 9 File: USPT Dec 19, 1995

US-PAT-NO: 5476992

DOCUMENT-IDENTIFIER: US 5476992 A

TITLE: In-situ remediation of contaminated heterogeneous soils

DATE-ISSUED: December 19, 1995

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Ho; Sa V. Creve Coeur MO Brodsky; Philip H. Creve Coeur MO

US-CL-CURRENT: 588/204; 204/515, 204/545, 204/549, 405/128.55, 405/130, 405/131

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw Desc Image

☑ 8. Document ID: US 5340376 A

L21: Entry 8 of 9

File: USPT

Aug 23, 1994

US-PAT-NO: 5340376

DOCUMENT-IDENTIFIER: US 5340376 A

TITLE: Controlled-release microbe nutrients and method for bioremediation

DATE-ISSUED: August 23, 1994

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Cunningham; John Tracy CA

US-CL-CURRENT: 71/6; 210/610, 435/262, 71/64.11, 71/903

Full Title Citation Front Review Classification Date Reference Sequences Attachments KMC
Draw, Desc Image

☐ 9. Document ID: US 5258303 A

L21: Entry 9 of 9

File: USPT

Nov 2, 1993

US-PAT-NO: 5258303

DOCUMENT-IDENTIFIER: US 5258303 A

TITLE: Bioremediation system and method

DATE-ISSUED: November 2, 1993

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Stenger; Raymond C. Freehold NJ 07728

US-CL-CURRENT: 435/262; 435/299.1

Full Title Citation Front Review Classification Date Reference Sequences Attachments

Draw, Desc Image

Generate Collection | Print

Terms	Documents
14 and L20	9

WEST

Generate Collection Print

L21: Entry 2 of 9 File: USPT Dec 4, 2001

DOCUMENT-IDENTIFIER: US 6325934 B1

TITLE: Enzyme and bacterial combination in a slowly dissolvable matrix for septic

tanks, grease traps and waste treatment

Brief Summary Text (9):

This invention relates to granules of a slowly dissolving, water soluble. thermoplastic matrix containing enzymes, <u>bacteria or bacteria/enzyme</u> combinations, and to their use in granular septic tank bioaugmentation preparations, grease trap cleaning formulations and other bioremediation agents.

Brief Summary Text (11):

Septic tanks and other biological treatment devices and facilities stabilize influent waste by removing biochemical oxygen demand (BOD), mineralizing carbon-containing compounds, digesting organic solids, and converting other compounds to chemical forms that are less damaging to the environment. It is important that organic components that might occlude, accumulate, and coat waste treatment, waste holding and waste conveying systems be dissolved and digested in an environmentally acceptable manner. Enzymes, <u>bacteria</u>, and <u>bacteria-containing</u> enzyme rearations are widely used in septic tank treatments, bioaugmentation agents, grease trap treatments, and bioremediation for other biological waste water processes. There is also a problem of sludge accumulation in lagoons and pits designed for the digestion of human and animal waste which needs a solution. Normally the granulated bacteria and enzymes are used as components of a comprehensive or diverse formulation which contains active ingredients compounded to enhance and accelerate the digestion of waste products. Preparations of this type are known as bioaugmenitation or bioenhancement agents. The three types of biological components of a bioenhancement agent, that is enzymes, bacteria or bacteria/enzyme combinations may be used in varying concentrations depending upon the situation. For example, some geographic areas regulate products in such manner that only viable bacteria containing additives may be used; free, active enzymes must be excluded from bioaugmentation products. In other areas combination products are used. Additionally, situations that are in food processing or medical facilities may require enzyme only (bacteria-free) products for waste treatment.

Brief Summary Text (19):

U.S. Pat. No. 5,275,943 DiTuro (Jan. 4, 1994) discloses at the abstract, "Tablets are formed that release components over time for biological degradation of organic matter such as sewage sludge, petroleum hydrocarbons, pesticides and herbicides. The tablets contain an inner-core of a dormant live microorganism, an inner-coating over the inner-core of water soluble hydroxypropyl methylcellulose or polypropylene glycol, an outer-layer over the inner coating of sodium sulfate coated sodium carbonate peroxyhdrate particles, and an outer-coating over the outer-layer of water soluble hydroxypropyl methylcellulose or polyethylene glycol. The inner core may contain a binder such as paraffin, gelatin and dextrose, and the outer-layer may contain additives such as enzymes, buffering agents, sugars and manganese dioxide as an oxidation catalyst. When the tablets are placed in an aqueous environment, layers of the tablet dissolve over time releasing components therein." At Column 1, lines 7 through 14 DiTuro discloses "This invention is directed to a non-toxic in situ method for the accelerated biological degradation of organic matter in the form of sewage sludge or petroleum hydrocarbons on the surface of aquatic objects submerged in bodies of saltwater, brackish, or freshwater". At col. 3, lines 21-27 DiTuro discloses "This invention proposes an in situ approach to help remediate this

problem. Its approach is one in which the ability of naturally occurring and/or seeded microorganisms to break down these contaminants is enhanced by the $\underline{\text{timed}}$ $\underline{\text{release}}$ of oxygen gas, via chemical reaction, and chemical additives such as $\underline{\text{buffering}}$ agents and enzymes."

Brief Summary Text (25):

The present invention relates to small granules of a slow release digesting composition which, in one embodiment, preferably sinks and is distributed to the entire bottom area of the digester to digest the sludge. In another embodiment, the granules are grease soluble and digest grease. Another embodiment, combines the two. The digesting composition of the present invention is preferably made by incorporating bacteria/enzyme combinations and other biologically active materials in a matrix in order to create a time sustained release of biologically active components including viable bacteria, enzymes, and other beneficial compounds such as nutrients into an aqueous waste environment. Instead of the combination, bacteria, or enzymes can be incorporated into the matrix separately, but this is not preferred. The enzymes initiate the digestion of the sludge and are active only for a day to a week depending upon the formulation, whereas the bacteria become a permanent digestive colony. The purpose of said invention is to render the active components efficiency in a waste water treatment, holding or conveying systems more economical and trouble-free.

Brief Summary Text (31):

To further control the rate of release of the bacteria and hydrolytic enzyme said surfactant is preferably combined with a non-toxic wax. This also increases the fat solubility of the granulated composition. The wax can be selected from the class comprising paraffin wax, wax of natural orgin such as beeswax or synthetic wax. The preferred wax is a paraffin wax. Paraffin wax is a hydrocabon or petroleum wax. The wax is preferably present in range of 1% to 50% of the weight of the total composition.

Brief Summary Text (48):

The preferred final granulated product has an average diameter of 0.5 to 5 mm and preferably 1 to 2.5 mm. The proportions of ingredients are not critical so long as there is a small but sufficient amount of slowly water soluble matrix to surround each enzyme and each bacteria to provide the required timed release.

Detailed Description Text (2):

Turning now to FIG. 1, there is shown the granules 1 of the present invention being introduced from a box 3 into a toilet 5. Flushing toilet 5 carries granules 1 through drain pipe 7 into septic tank 9. The granules 1 settle through grey water 11 dispersing uniformly across the top 13 of sludge layer 15. The granules 1 will sink into sludge layer 15 with time. The grey water exits the septic tank 9 through discharge line 17 after passing barrier 19. In FIG. 2, FIG. 3 and FIG. 4, the diameter or thickness of the granule is depicted by the reference numerals 21, 22, 23 and 25. ache terms "diameter" and "thickness" are used interchangeably, and are not critical. All that is required is that the granules be able to pass through the toilet 5 and uniformly disperse in sludge layer 15.

Detailed Description Text (12):

Upon liquefaction, the surfactant was physically blended (homogenized) with a mixer or by hand with the enzyme and limestone granules until a uniform consistency was achieved. The coating/enzyme was then allowed to cool and solidify at room temperature (approximately 26.degree. C.). This allowed a uniform Layer of surfactant to surround the enzyme and limestone granules.

Detailed Description Text (16):

Upon liquefaction, the surfactant wax combination was physically blended (homogenized) with a mixer with the enzyme until a uniform consistency was achieved. The coating/enzyme combination was then allowed to cool and solidify at room temperature (approximately 26.degree. C.). This allowed a uniform layer of surfactant to surround the enzyme granule. Upon granulation this composition is useful to break down animal fats in granule traps and sewer lines.

Detailed Description Text (20):

Upon liquefaction, the surfactant wax combination was physically blended (homogenized) with a mixer with the enzyme and limestone granules until a uniform consistency was achieved. The coating/enzyme and limestone was then allowed to cool and solidify at room temperature (approximately 26.degree. C.). This allowed a uniform layer of surfactant to surround the enzyme and limestone granules.

WEST

Generate Collection Print

L21: Entry 3 of 9 File: USPT Aug 17, 1999

DOCUMENT-IDENTIFIER: US 5939086 A

TITLE: Compositions and methods for reducing the amount of contaminants in aquatic

and terrestrial environments

Abstract Text (1):

The invention provides contaminant-reducing agent <u>delivery compositions that are useful for the control</u> of organic or inorganic contaminants in aquatic or terrestrial environments. The compositions comprise one or more superabsorbent solid organic polymers and at least one contaminant-reducing agent. These superabsorbent polymers act as the primary carriers of one or more contaminant-reducing agents effective for the control of organic or inorganic contaminants in aquatic or terrestrial environments. Contaminant-reducing agents include film-forming agents, microbial agents, nutrient agents, and mixtures thereof. The invention also provides a method to entrap and accumulate organic and inorganic contaminants in one or more superabsorbent polymer compositions containing no contaminant-reducing agent. The invention also provides a method to entrap water in superabsorbent polymers to activate natural and applied microbial and nutrient contaminant-reducing agents in terrestrial environments.

Brief Summary Text (5):

Different approaches to reducing the concentration of environmental contaminants have been explored. One such approach is bioremediation, which is the use of living organisms to break down organic and inorganic contaminants into more basic components, remove environmental pollutants, restore contaminated sites, and prevent the accumulation of further pollutants. Generally, bioremediation is accomplished using bacteria, although other microorganisms, namely fungi and algae, have been used. See, for example, Ronald M. Atlas, Chemical & Engineering News, Apr. 3, 1995, pp. 32-42. There still exists a need, however, to reduce the amount of organic and inorganic contaminants in aquatic and terrestrial environments.

Brief Summary Text (7):

The present invention provides a contaminant-reducing composition comprising a contaminant-reducing agent and a superabsorbent solid organic polymer for delivery of the contaminant-reducing agent, wherein said composition is capable of reducing the amount of an organic or inorganic contaminant in an aquatic or terrestrial environment. The composition may be solid or liquid. If liquid, the composition is preferably capable of being sprayed, pumped, or injected. The composition may also be a controlled-release composition.

Brief Summary Text (8):

Preferably, the superabsorbent polymers are hydrophilic acrylamide and acrylate polymers which then act as matrices for the controlled release (e.g., fast, slow, pulsed, delayed) of the contaminant-reducing agent employed, according to zero-order, first-order, or square-root-of-time kinetics. More preferably, the superabsorbent polymer of the present invention is a combination of a polysaccharide and an organic monomer, oligomer, polymer, copolymer, terpolymer or tetrapolymer. Most preferably, the superabsorbent polymer of the present invention is the product of grafting amylopectin with acrylonitrile. Such superabsorbent polymers can also used by themselves (i.e., without the contaminant-reducing agent) to entrap and accumulate organic and inorganic contaminants, thereby preventing leaching or movement and further damage to areas of the environment that are far removed from the initial point(s) of contamination. The compositions may further include one or

more non-toxic adjuvants or diluents.

Brief Summary Text (10):

The present invention also provides a method of reducing the amount of organic or inorganic contaminants in an aquatic or terrestrial environment. The method involves applying the contaminant-reducing composition described above to the contaminated site. Upon application, the contaminant-reducing agent impregnated in the superabsorbent polymer is released into the environment. Preferably, this release occurs in a controlled manner. Release of the contaminant-reducing agent can occur, for example, via a water-activated swelling-controlled diffusion process, and/or the biodegradation (i.e., microbial) or degradation (e.g., decomposition via ultraviolet light) of the polymer.

<u>Detailed Description Text</u> (2):

Surprisingly, it has been found that certain superabsorbent polymers are useful as matrices for contaminant-reducing agents effective for reducing the concentration of organic and inorganic contaminants, in aquatic or terrestrial environments, with a single application or multiple applications of a solid or liquid formulation. These compositions may be in controlled-release form.

Detailed Description Text (3):

Superabsorbent polymers, including starch graft copolymers, are well known in the art. See, for example, those described in U.S. Pat. No. 4,375,535 (Kightlinger et al.), and U.S. Pat. No. 4,497,930 (Yamasaki et al.). These polymers have been used as adhesives, flocculents, and as water-retaining materials for agricultural or sanitation uses. Superabsorbent polymers have also been used alone in pest management applications or in combination with pesticides or insecticides as matrices for their release in aquatic or terrestrial environments. However, the advantages of using this class of polymers as contaminant-entrapment matrices, as matrices for water-activation of contaminant-reducing agents, and as matrices for the controlled release of active agents in aquatic or terrestrial environments to control organic or inorganic contaminants have gone unrecognized until the present invention.

Detailed Description Text (5):

It has been observed that certain superabsorbent polymers that are impregnated with one or more contaminant-reducing agents have the ability to swell in water and/or biodegrade/degrade, and thereby release the substance(s) contained therein. Furthermore, it has been observed that the superabsorbent polymers that swell when exposed to water also have the ability to reform or contract to a consistency similar to their original form when evaporation has caused the water to be removed from the polymer matrix, and then reswell with additional exposure to water. This ability to be functional after repetitive periods of exposure to water is advantageous for pretreatment and/or prolonged controlled release applications. Certain superabsorbent polymers also have the ability to entrap and accumulate, i.e., concentrate, contaminants to facilitate their removal. Also, certain superabsorbent polymers (e.g. starch-graft) applied to terrestrial habitats can promote the degradation of contaminants by natural or applied microorganisms by entrapping water in surface or subsurface areas, and thereby provide higher and prolonged moisture levels and added nutrients (i.e., starch) for enhanced microbial growth and development. Similarly, the water entrapped within superabsorbent polymers can also activate and prolong the activity of natural or applied nutrient contaminant-reducing agents.

Detailed Description Text (21):

As used herein, the term "microbial agents" is meant to include microorganisms that enhance biodegradation processes. These microorganisms include bacteria, fungi, and algae, or combinations of these microorganisms, for example. Furthermore, as used herein, "biodegradation" means the chemical alteration and breakdown of a substance to usually smaller products caused by microorganisms and their enzymes. Contaminants that are biodegradable include any compound that can be microbially mineralized into carbon dioxide, water, ammonia and/or chloride, or that can be transformed into a non-hazardous intermediate. "Bioremediation" refers to the use of living organisms, primarily microorganisms, to convert harmful organic or inorganic contaminants into carbon dioxide, water, cell tissue, and energy, to remove pollutants from the

environment, to restore contaminated sites, and to prevent further pollution from accumulating. Generally, contaminants amenable to bioremediation include: petroleum hydrocarbons, such as gasoline, diesel, fuel oil, crude oil, refinery sludges; aromatics, such as benzene, toluene, xylene, ethylbenzene, styrene; polynuclear aromatic hydrocarbons, such as naphthalene, phenanthrene, pyrene, benzo[a]pyrene; alcohols, such as isopropanol, ethanol, ethylene glycol, t-butanol; carbohydrates, animal fats and greases; detergents; ketones, such as acetone, methyl ethyl ketone, phenols, such as chlorophenol, pentachlorophenol; polychlorobiphenyls; phthalates; chlorinated solvents, such as methylene chloride, ethylene dichloride.

<u>Detailed Description Text</u> (34):

C. Controlled Release Contaminant-Reducing Agent Delivery Composition

Detailed Description Text (36):

In particular, the present invention is directed toward a method of formulating one or more superabsorbent polymer(s) with one or more contaminant-reducing agents, with or without water or other additives, into compositions such as solid powders, dusts, granules, agglomerates, pellets, briquets, extrusions, laminates, or composites, or into sprayable, pumpable, or injectable, variable-viscosity water or oil base gel or semi-gel like formulations that can release one or more active ingredients to simultaneously or concurrently control a variety of inorganic or organic contaminants with a single or multiple application of a solid or liquid single or multi-product formulation. Preferably, the release occurs in a controlled manner.

Detailed Description Text (37):

Impregnation of superabsorbent polymers with fatty alcohol film-forming agents such as POE-2-isostearyl alcohol or sorbitan monooleate appear to delay or slow down the rate of water absorption of superabsorbent polymers such as SUPER SORB or WATER LOCK G-100, thereby providing another useful mechanism for slow or controlled release of contaminant-reducing agents.

Detailed Description Text (38):

The slow or controlled release process could be further modified or delayed by the degree of compaction of the powdered or flaked superabsorbent polymer and superabsorbent polymer/contaminant-reducing agent formulations, by varying the size of an orifice or the number of orifices in a container into which the formulation is placed, by varying the concentration of film-forming agent, by varying the concentration of different types of superabsorbent polymers, and by adding one or more binders. For example, using one or more cationic, anionic, or nonionic surfactants or surface active agents in the composition can regulate the rate and duration of delivery (i.e., increase or decrease) of the contaminant-reducing agent from the superabsorbent polymer matrix. Additionally, a coating can be utilized to alter the swelling potential of the superabsorbent polymer, and thereby slow the rate of release of the contaminant-reducing agent from the superabsorbent polymer matrix. The coating can be hydrophobic or have varying degrees of water solubility.

Detailed Description Text (39):

It should be noted that certain salts (e.g., alkali metal halides such as NaCl) have been shown to reduce the swelling of the superabsorbent polymer matrix when introduced into water. This can have an impact on the contaminant-reducing agent delivery composition's ability to swell and control the target contaminant. Therefore, it is possible to utilize certain salts in superabsorbent polymer-based compositions as another mechanism to adjust the controlled-release rate and controlled-release profile of the contaminant-reducing agents contained therein. The rate of release will be further modified by the salinity of the environment. For example, certain superabsorbent polymers swell less in sea water, so the rate of release is slower.

Detailed Description Text (53):

A series of bioremediation bioassays were conducted in three water qualities to evaluate the efficacy of granular controlled delivery contaminant-reducing microbial compositions composed of a starch grafted sodium polyacrylate superabsorbent polymer matrix (SANWET IM-1500 LP), an acrylic acid, co-polymer release-rate regulator (PEMULEN TR-1; The B. F. Goodrich Company, Cleveland, Ohio) and a petroleum-degrading bacterial formulation (ABR Diesel Blend of viable bacterial

cultures on a wheat bran base; code: 9577; Sybron Chemicals Inc., Palm Harbor, Fla.), in reducing the amount of aquatic contamination of No. 2 diesel fuel. Superabsorbent polymer granules were loaded in an aqueous formulation of bacterial and release rate regulator according to the following microsponging and entrapment procedure: 0.375 g (0.1% w/w) PEMULEN TR-1 was added to 337.12 g (89.9% w/w) water purified by reverse osmosis filtration (RO) in 500 ml NALGENE bottles and vigorously mixed on a STROKEMASTER paint shaker for approximately 30 minutes. Next, 37.5 g (10% w/w) ABR Diesel Blend was then added to the aqueous formulation and mixing was continued for an additional 60 minutes. Subsequently, 5.0 g (w/w) SANWET IM-1500 LP superabsorbent polymer granules were then added into the aqueous bacterial formulation and mixed for an additional 30 minutes to load the granules with the aqueous formulation ingredients. Swollen (hydrated) granules containing the formulation ingredients were rinsed with approximately 1000 ml RO water on a 10 mesh sieve to remove excess coating. Swollen granules were dried in a low-humidity room maintained at approximately 27-38% relative humidity (RH) and 24-26.degree. C. for 96 hours. Dry, dehydrated granules were placed into 40 ml glass vials and stored until use. Granule composition was approximately 59.0% (w/w) ABR Diesel Blend, 0.6% (w/w) PEMULEN TR-1, and 40.4% (w/w) SANWET IM-1500 LP superabsorbent polymer.

Detailed Description Text (55):

Granular controlled release SANWET IM-1500 LP superabsorbent polymer-base nutrient compositions utilized in conjunction with the bacterial composition of Example 3 consisted of a balanced blend of nitrogen, phosphorus, and micronutrients (BI-CHEM Accelerator II Special; code: 9546; Sybron Chemicals Inc., Palm Harbor, Fla.) and a release-rate regulator (PEMULEN TR-1). Superabsorbent polymer granules were loaded in an aqueous formulation of nutrients and a release-rate regulator ingredients. The following microsponging and entrapment procedure was utilized to formulate controlled delivery granules for bioassay: 0.375 g (0.1% w/w) PEMULEN TR-1 was added to 337.12 g (89.9% w/w) water purified by reverse osmosis filtration (RO) in 500 ml NALGENE bottles and vigorously mixed on a STROKEMASTER paint shaker for approximately 30 minutes. Further, 37.5 g (10% w/w) BI-CHEM Accelerator II Special was then added to the aqueous formulation and mixed for an additional 60 minutes. Next, 5 g (w/w) SANWET IM-1500 LP superabsorbent polymer granules were then added to the water-base nutrient formulation. Swollen (hydrated) granules were rinsed with approximately 1000 ml RO to remove excess coating. Swollen granules were dried in a low-humidity room at approximately 27-38% relative humidity and 24-26.degree. C. for 96 hours. Dry, dehydrated granules were placed into 40 ml glass vials for storage until use. Granule'composition was approximately 62.1% (w/w) BI-CHEM Accelerator II Special, 0.6% (w/w) PEMULEN TR-1, and 37.3% (w/w) SANWET IM-1500 LP superabsorbent polymer.

Detailed Description Text (57):

A series of comparative bioremediation granule-transfer bioassays were conducted to determine the efficacy of controlled delivery superabsorbent polymer-based granular formulations composed of contaminant-degrading bacterial microorganisms and nutrients in reducing the level of petroleum contaminants in aquatic habitats having three types of water quality. The target petroleum contaminant was No. 2 diesel fuel.

Detailed Description Text (60):

Two granular application rates for each bacterial and nutrient composition were evaluated in the 3 water qualities containing the petroleum contaminant. Rates were applied to achieve bacterial levels of approximately 10.sup.5 -10.sup.6 cfu (colony forming units)/ml. A low granular application rate consisted of applying 0.35 g (approximately 292 granules at 0.0012 g/granule) of SANWET IM-1500 LP superabsorbent polymer granules loaded with ABR Diesel Blend (i.e., 0.2 g ABR Diesel bacterial cultures) and 0.65 g (approximately 342 granules at 0.0019 g/granule) of SANWET IM-1500 LP superabsorbent polymer granules loaded with BI-CHEM Accelerator II Special nutrient complex to the surface of each crystallizing dish containing 25,000 ppm No. 2 diesel fuel on RO, 50% seawater, and 100% seawater. At this application rate, controlled delivery ABR Diesel bacteria granules contained approximately 2.times.10.sup.6 cfu/ml when applied to the petroleum-contaminated aquatic habitats.

A high granular application rate consisted of applying 0.70 g (approximately 584 granules at 0.0012 g/granule) of SANWET IM-1500 LP superabsorbent polymer granules loaded with ABR Diesel Blend (i.e., 0.4 g ABR Diesel bacterial cultures) and 1.3 g (approximately 684 granules) of SANWET IM-1500 LP superabsorbent polymers loaded with BI-CHEM Accelerator II Special nutrient complex to the surface of each crystallizing dish containing 25,000 ppm No. 2 diesel fuel on RO, 50% seawater, and 100% seawater. At this application rate, controlled delivery ABR Diesel bacteria granules contained approximately 4.times. 10.sup.6 cfu/ml when applied to the petroleum-contaminated aquatic habitats.

Detailed Description Text (64):

Duration of effective controlled delivery of satisfactory concentrations of ABR Diesel Blend bacterial cultures and BI-CHEM Accelerator II Special was determined by transferring the hydrated bacteria and nutrient loaded granules to new test chambers (i.e., crystallizing dishes) containing new water and No. 2 diesel fuel, and evaluating the rate and duration of biodegradation of the diesel resulting from the release of bacteria from the granules. Comparative qualitative evaluations were based on the visual degradation (i.e., emulsification and reduction in size) of the surface layer of the No. 2 diesel fuel in all water quantities on a daily basis. Quantitative evaluations were based on diesel degradation in the high bacterial and nutrient application rate. Comparative post-treatment analyses of the concentration of diesel in tests and controls were only conducted in 100% seawater. Analyses of the average Total Petroleum Hydrocarbons (TPH) at various posttreatment intervals were conducted in pooled samples of the 3 water qualities according to EPA protocol 418.1 (Sybron Chemicals Inc.). At these posttreatment periods, the bacterial and nutrient granules were removed from each water quality, rinsed through a basket strainer, and transferred to new test chambers containing 495.0 g of RO, 50% seawater, or 100% seawater, 3 g BI-CHEM Accelerator II Special, and 5 g No. 2 Diesel fuel (i.e., 10,000 ppm). A series of granule transfers were evaluated (e.g., Transfers #1, 2, and 3).

Detailed Description Text (65):

Visual observations of the petroleum layer indicated a gradual emulsification/gellying and reduction in the size of the layer of No. 2 diesel fuel when compared to controls. The rate of biodegradation appeared to be slightly faster in the standards when compared to the superabsorbent polymer granular compositions; however, this was attributed to the lower level of free bacterial cultures initially present in the water column due to the slow rate of release of the bacterial from the superabsorbent polymer granules. Similar visual signs of biodegradation of the No. 2 diesel fuel layer were recorded in all granule transfer tests. Comparative observations indicated that the speed of degradation was directly related to the application rates of the loaded superabsorbent polymer granules, i.e., degradation of the diesel layer appeared to be about twice as fast in tests at the high bacterial and nutrient rates when compared to tests conducted at the lower rates.

Detailed Description Text (67):

IM-1500 LP Superabsorbent Polymer-base bacteria and nutrient granules transferred from the 25,000 ppm diesel-contaminated water qualities at 21-days posttreatment to the 10,000 ppm No. 2 diesel fuel water qualities were observed to be effective in slowly degrading the petroleum-contaminant in the 3 water qualities. Emulsification of the diesel layer resulting in a gellying and reduction in the diameter of the surface layer of diesel was observed to slowly increase over the initial 21-day test period. A second series of analyses (Transfer #1) of the diesel contamination in the 3 water qualities treated with the controlled delivery superabsorbent polymer-base bacterial and nutrient granular compositions at 19 days posttransfer (i.e., 40 days posttreatment) resulted in average TPH levels of 7100, 455, and 488 mg/l in RO, 50% seawater, and 100% seawater tests, respectively. The TPH levels in the controls were 9,810-10,080 mg/l. Similar visual signs of biodegradation of the diesel layer were observed over the 19 day posttransfer period in the 3 water qualities.

<u>Detailed Description Text</u> (68):

It should be noted that visual observations of the third transfer at 14 days post-transfer of the superabsorbent polymer granules (i.e., 54 days posttreatment) containing the bacterial and nutrients to new diesel-contaminated (i.e., 10,000 ppm) water (RO, 50% seawater, and 100% seawater) indicated that the rate of

biodegradation of the surface <u>layer</u> of diesel was comparable to the second transfer. Granules were still active when the tests were terminated, suggesting that the starch-grafted superabsorbent polymer granules were still effective in releasing satisfactory levels of the diesel-degrading bacterial and nutrients for a prolonged period. In addition, it was possible that the starch-based granules also served as "mini-fermenters" for regenerating bacteria within the starch-grafted matrix for slow-release into the water. Different degrees of biodegradation of the granules were observed in the transfers.

Detailed Description Text (70):

In general, results of bioassays indicated that superabsorbent polymer granules can be used as matrices for the prolonged controlled release of bacteria and nutrients used for bioremediation of petroleum contaminants in aquatic environments having a variety of water qualities. Controlled release of the contaminant-reducing agents appeared to be via a swelling controlled diffusion process and gradual biodegradation of the superabsorbent polymer matrices. The rate of petroleum biodegradation was directly related to the application rate of bacteria and nutrient granules. Bioassay results further suggested that the dense controlled-release granular compositions would produce petroleum contaminant-reducing efficacy in shallow water, but would be required to float at or near the surface of the water to deliver the contaminant-reducing agent(s) in proximity to the surface contaminant in deep water habitats such as oceans. Results also suggested that the starch-grafted superabsorbent polymer granules may serve as a nutrient source for regeneration of the bacteria for prolonged controlled release.

Detailed Description Text (72):

A series of terrestrial bioremediation bioassays were conducted in soil to evaluate the efficacy of powdered controlled delivery admixture formulations composed of a starch-g-poly(2-propenamide-co-2-propanoic acid, sodium salt) superabsorbent polymer (WATER LOCK A-140), a No. 2 diesel fuel-biodegrading bacterial formulation (i.e., ABR Diesel Blend of viable bacterial cultures on a wheat bran base; code: 9577), and a nutrient blend (i.e., BI-CHEM Accelerator II Special; code: 9546) in reducing the levels of a petroleum contaminant on land. The target contaminant utilized in these terrestrial bioremediation bioassays was No. 2 diesel fuel (i.e., 25,000 ppm/petri dish habitat). Tests were conducted in uncontaminated field collected soil used for fill at the Lee County Mosquito Control District. Tests were replicated 3 times.

Detailed Description Text (74):

In the protocol for superabsorbent polymer-base compositions, a premixed (5 minutes) formulation of 0.07 g (0.01% w/w) ABR Diesel Blend of viable bacterial cultures on a wheat bran base (code: 9577), 12.60 g (1.15% w/w) BI-CHEM Accelerator II Special nutrient blend (code: 9546), and 12.67 g (1.15% w/w) superabsorbent polymer powder (WATER LOCK A-140) was thoroughly blended into the diesel-contaminated soil with a KITCHENAID KSM 90 mixer for approximately 7 minutes to achieve a homogeneous mixture of the components. Next, 153.62 g of this superabsorbent polymer/soil composition was added to each 100.times.15 mm glass petri dishes and lightly hand compacted to level the soil-controlled release admixture. Throughout the soil surface and subsurface, 5 ml RO water was pipetted to provide a moist soil environment and swollen superabsorbent polymer matrices of bacteria and nutrients throughout the soil. The soil was remoistened with 5 ml RO water at 3-day intervals and aerated by turning the soil with a spatula and re-leveled at 1-3 day intervals. Each petri dish in this series contained approximately 1.times.10.sup.5 cfu/g soil (approximately 0.01 g ABR Diesel bacteria cultures on a wheat bran base), 1.80 g BI-CHEM Accelerator II Special nutrient complex, 1.81 g WATER LOCK A-140 superabsorbent polymer powder, and 3.75 g (i.e., 25,000 ppm) No. 2 diesel fuel.

Detailed Description Text (77):

Bioassay room temperature and humidity ranged from 26-33.degree. C. and 74-86% relative humidity, respectively. Biodegradation of the No. 2 diesel fuel in soil with the superabsorbent polymer and non-superabsorbent polymer-base powdered compositions was evaluated on a quantitative basis. Effectiveness of the petroleum-reducing, controlled release powdered formulation of ABR Diesel Blend bacteria, BI-CHEM Accelerator II Special nutrient blend, and WATER LOCK A-140 superabsorbent polymer matrices in biodegrading the No. 2 diesel contaminated throughout the soil was based on quantitative analyses of the concentration (mg/kg)

of Total Petroleum Hydrocarbons (TPH) of pooled soil subsamples at various posttreatment intervals (EPA test SW-846 9071; Sybron Chemicals Inc.). This data was compared to the TPH levels in non-superabsorbent polymer standards and controls.

Detailed Description Text (78):

Duration of effective controlled delivery of satisfactory concentrations of bacteria and nutrients from the WATER LOCK A-140 superabsorbent polymer matrices throughout the soil was determined by transferring at 21 days posttreatment (Transfer #1) one-half of each superabsorbent polymer-base and non-superabsorbent polymer-base replicate (i.e., 75 g) containing the remaining (i.e., partially biodegraded) diesel-contaminated soil and nutrient/ABR Diesel Blend to new glass petri dishes containing an equivalent amount of clean soil (i.e., 75 g), 1.5 g (10,000 ppm) No. 2 diesel fuel, and 1.8 g nutrients (BI-CHEM Accelerator II Special). All components were blended together with a KITCHENAID KSM 90 mixer for approximately 5 minutes to assure a homogeneous mixture before being transferred into the new test chambers.

<u>Detailed Description Text</u> (79):

TPH analyses of soil containing No. diesel fuel (25,000 mg/kg), and a superabsorbent polymer-base (i.e., WATER LOCK A-140) ABR Diesel Blend and BI-CHEM Accelerator II Special powdered composition resulted in an average TPH level of 10,400 mg/kg at 21 days posttreatment. At this time period, approximately 58% of the No. 2 diesel fuel was biodegraded by the ABR Diesel Blend. Diesel-contaminated soil treated with the non-superabsorbent polymer-base composition (i.e., the standard) contained an average TPH level of 11,300 mg/kg, or a diesel No. 2 reduction of approximately 55%. These results indicated that the increased biodegradation efficacy observed with the superabsorbent polymer-base bacteria and nutrient compositions was due to the slower rate of evaporation that would be expected from water entrapped within the superabsorbent polymer matrices within the soil, when compared to non-encapsulated or free water within the soil. The higher degree of soil moisture was presumed to provide a more optimal environment for bacterial growth. Perhaps the starch portion of the superabsorbent polymer-base controlled delivery composition also added an additional nutrient source for increased bacterial growth.

Detailed Description Text (80):

Analyses of the controlled delivery superabsorbent polymer-base soil composition that was transferred at 21 days posttreatment (Transfer #1) to an admixture of nutrient-enriched soil contaminated with an additional 10,000 ppm No. 2 diesel fuel at 18 days posttransfer (i.e., at 39 days posttreatment) resulted in an average TPH concentration of 3780 mg/kg. Transfer of the non-superabsorbent polymer-base composition resulted in an average concentration of TPH of 4810 mg/kg. In general, results of comparative transfer bioassays indicated that the starch-grafted superabsorbent polymer matrices were effective in prolonging the release of entrapped petroleum-reducing bacteria and nutrients, while providing a more optimum soil moisture level and possible nutrient source for enhanced microbial activity.

Detailed Description Text (82):

A series of terrestrial bioremediation bioassays were conducted on sand to evaluate the efficacy of controlled delivery semi-viscous, liquid (i.e., water based) compositions of a starch-g-poly(2-propenamide-co-2-propanoic acid, potassium salt) superabsorbent polymer (WATER LOCK B-204) and a petroleum-degrading bacterial culture formulation (BIOTRACK DOL; code: 9690) in effectively reducing the level of surface petroleum contamination on land. The substrate utilized for this terrestrial bioassay series was TEXBLAST sand-blasting sand. The target contaminant was No. 2 diesel fuel.

<u>Detailed Description Text</u> (84):

Bioassay room temperature and humidity ranged from 26-33.degree. C. and 74-86% relative humidity, respectively. Biodegradation of the No. 2 diesel fuel on the surface of the sand with the superabsorbent polymer-base and non-superabsorbent polymer-base formulations was evaluated on a quantitative basis. Effectiveness of the petroleum-reducing controlled delivery, variable-viscosity formulation of BIOTRACK DOL bacterial composition and WATER LOCK B-204 superabsorbent polymer matrices in biodegrading the No. 2 diesel fuel contaminated on the surface of the sand was based on analyses of the concentration Total Petroleum Hydrocarbons (TPH) of sand subsamples at 21 days post-treatment. This data was compared to the

concentration of TPH in non-superabsorbent polymer-base standards and controls.

Detailed Description Text (85):

Duration of effective controlled delivery of satisfactory levels of petroleum-degrading bacteria from the WATER LOCK B-204 superabsorbent polymer matrices on the No. 2 diesel-contaminated soil surface was determined by transferring at 21 days posttreatment one half of each superabsorbent polymer-base and non-superabsorbent polymer-base replicate (i.e., 75 g) containing the remaining (i.e., partially biodegraded) diesel-contaminated sand and BIOTRACK DOL to the surface of the sand in new glass petri dishes containing an equivalent amount of clean sand (i.e., 75 g) that was surface-coated with 1.25 g No. 2 diesel fuel (i.e., 10,000 ppm).

Detailed Description Text (88):

Three different superabsorbent polymers were formulated with a film-forming or bacterial contaminant-reducing agent and inert controlled release-rate regulator ingredients to evaluate the effect of superabsorbent polymer type and inert formulation components on the concentration of active contaminant-reducing agent loaded into the granules, and the profile of release of the components from the granules into an aquatic habitat: Superabsorbent polymer granules utilized in these evaluations were a potassium polyacrylate, lightly crosslinked (ARIDALL 11250), a starch grafted sodium polyacrylate (SANWET IM-1500 LP), a partial sodium salt of a crosslinked polypropenoic acid (Dow XU 40346.00 Super Absorbent Polymer), a poly (sodium acrylate) homopolymer (SS Superabsorbent Polymer), a crosslinked polyacrylamide copolymer (ALCOSORB AB3C), a crosslinked potassium/polyacrylamide copolymer (STOCKOSORB 310 K), or a copolymer of acrylamide and sodium acrylate (AQUASORB PR-3005). Release-rate regulators included inert formulating materials that can retard or accelerate the rate of delivery or diffusion of the active contaminant-reducing agent(s) from the superabsorbent polymer granules; depending on the propensity of the active agents to leach from or be bound within the polymer matrices. Several nutrient formulations with no release-rate regulators were also evaluated. Non-limiting examples of release-rate regulators utilized in the admixtures included activated carbon (UCAR Carbon Co., Inc), hydrophilic silica (DEGUSSA FK 500 LS), acrylic acid copolymers (PEMULEN IR-1 or PEMULEN TR-2), sulfonated surfactants (sodium di-isopropyl naphthalene sulfonate: MORWET IP Powder; sodium mono and di-methyl naphthalene sulfonate: MORWET M Powder; or sodium alkyl aryl sulfonate: MORWET 3008 Powder), or a fatty alcohol polyethylene glycol ether (MARLIPAL 1618/25 P6000 powder). Contaminant-reducing agents included in the formulations were nutrient complexes (BI-CHEM Accelerator II Special), bacteria cultures that were unfiltered or filtered to remove large bran particles (ABR Diesel Blend; code: 9577 or ABR Gasoline Blend; code: 9350), or film-forming agents (sorbitan monooleate or a mixture of 75% sorbitan monooleate/25% 2-ethyl butanol). Microsponging and entrapment techniques were used to load the active and inert components in an aqueous medium into the superabsorbent polymers according to a 0.5, 1, or 3 hour aqueous admixing procedure (i.e., depending on the swelling potential the superabsorbent polymer granules) that is generally described in Example 4. The loading rates that were observed when superabsorbent polymer granules were admixed with an aqueous formulation of 10% contaminant-reducing agent and 1-10% inert formulation components (i.e., 1% MORWET products, 0.5% carbon and 0.1% silica for sorbitan monooleate-base compositions; or 10% MORWET or MARLIPAL products or 0.1% PEMULEN TR-1 for BI-CHEM Accelerator II nutrient compositions; or 0.1% PEMULEN TR-1 or TR-2 for ABR Gasoline Blend bacteria cultures; or 0.1% PEMULEN TR-1 for ABR Diesel Blend bacteria cultures) were as follows:

Detailed Description Text (158):

Results of formulation studies indicated that the concentration of film-forming and bacterial contaminant-reducing agents loaded into the superabsorbent polymer granules via an aqueous microsponging procedure was a function of the type of superabsorbent polymer and/or the inert release-rate regulators utilized in the admixtures. Release-rate regulators also functioned as loading-level regulators. A series of visual demonstrations were designed to evaluate the effect of the superabsorbent polymer and the release-rate regulators on the controlled release of the petroleum contaminant-reducing agents from the superabsorbent polymer granules.

<u>Detailed Description Text</u> (161):

Duration of controlled release of film-forming contaminant-reducing agents from floating agglomerated superabsorbent polymer-base compositions and devices was evaluated in 5 gallon plastic buckets containing 4 gallons of 100% seawater (INSTANT OCEAN) and/or in experimental fresh-water ponds (50.times.50 ft.) having a variety of natural vegetation and invertebrate and vertebrate organisms. Film-forming agents utilized in the tests were sorbitan monooleate, a mixture of 75% sorbitan monooleate/25% 2-ethyl butanol, or POE(2)isostearyl alcohol. WATER LOCK A-140 superabsorbent polymer was formulated in the agglomerated compositions used in the bioassays and field evaluations; however agglomerated compositions were also prepared using Water Lock A-100, A-120, SUPER SORB, FAVOR CA100, and AQUA KEEP J-500.

<u>Detailed Description Text</u> (162):

Agglomerated compositions of WATER LOCK A-140 and film-forming agents were prepared in the following manner: 100 g sorbitan monooleate, 75 g sorbitan monooleate/25 g 2-ethyl butanol, or 100 g POE(2) isostearyl alcohol were added to 300 g acetone in a stainless steel bowl and blended with a KITCHENAID KSM 90 mixer (wire whip attachment) for 5 minutes on #2 speed. While mixing, 100 g WATER LOCK A-140 superabsorbent polymer powder was slowly added into a film-forming agent. Mixing was continued until the acetone had been driven off and the powdered composition was essentially flowable (approximately 2-3 hours). Next, 15 g of powdered superabsorbent polymer/film-forming agent composition was tightly packed into preformed, water soluble 2 mil polyvinyl alcohol bags (MONOSOL 8000 Series) and heat (sealed. The encapsulated compositions were cured in a room maintained at ca 27.degree. C. and 80% relative humidity for approximately 96 hours before being transferred to a drying room maintained at approximately 27.degree. C. and 27-38% relative humidity for an addition 96 hours. 30 or 40 mesh nylon netting was then tightly or loosely wrapped around each bag to allow for different degrees of water-activated superabsorbent polymer swelling or expansion. Tightly fitted bags measured approximately 38.times.51 mm while loose fitting mesh bags were approximately 76.times.102 mm. A small cork or plastic float was attached to each end of a mesh bag to assure that the agglomerated compositions would be in contact with and just below the surface of the water in proximity to a floating contaminant. Powdered superabsorbent polymer-base film-forming agent compositions were also agglomerated into briquets or disquets by hand compaction of the compositions at various pressure in plastic tissue embedding molds (PEEL-A-WAY) or in plastic petri dishes (approximately 35.times.10 mm). 1:1 powdered controlled delivery compositions of superabsorbent polymers and ABR Diesel Blend or ABR Gasoline Blend or ABR Hydrocarbon Blend or BI-CHEM Accelerator II Special, with or without one or more release-rate regulators, were agglomerated and optionally encapsulated within water-soluble polyvinyl alcohol bags, or hand compacted into briquets or disquets as described above. Similar powdered compositions containing joint or multiple combinations of products such as 1:1:1 or 1:1:1:1 admixtures of a superabsorbent polymer, an ABR bacterial culture, a nutrient complex, and/or film-forming agent were agglomerated into a variety of matrix forms. By aligning the floating or submerged compositions in various arrangements and combinations, e.g., in layers, in tandem, linked together in a chain-type configuration, etc., one or more contaminants could be simultaneously contained and/or controlled for prolonged periods. Granular controlled-release compositions could be utilized in the same fashion. These controlled-delivery compositions would also have significant applications for the control of surface or subsurface contaminants in terrestrial habitats. Procedures for admixing, curing, drying, and hardening of the compositions are described above and in the examples.

Detailed Description Text (163):

Results of tests in fresh water ponds (approximately 240-280 umhos/cm) dusted with talc (i.e., to visualize spreading of the film-forming agents) indicated that the controlled release rate of a film-forming agent from the tight mesh bag was slower than from the loose mesh bag. Duration of film-forming agent persistence (i.e., film pressure) was monitored with ADOL (oleyl alcohol) indicator oil. Daily water temperature was approximately 35.degree. C. Mesh size also affected the rate of delivery by restricting or slowing the release of expanded superabsorbent polymer-base components into the water. Bioassay results in buckets containing 100% seawater indicated that release of the film-forming agents from the superabsorbent polymer compositions was significantly slower than observed in the fresh water due

to the general inhibition of superabsorbent polymer swelling in high salinities. Similar trends in release due to mesh tightness and mesh size were also observed.

Detailed Description Text (164):

In general, these observations suggested that various devices could be fabricated that could regulate the rate of delivery of the contaminant-reducing or controlling agent by restricting the polymer swelling potential and/or surface area of the polymer in contact with the water, for example, by varying the size and number of openings or pores in a hollow, floating or submerged device. Further modification in release rate could be obtained by selecting superabsorbent polymers with different swelling and biodegradation parameters and/or by admixing various release-rate regulators ingredients into the formulation. Low pressure and high pressure compaction techniques and the use of various binders and hydrophobic or insoluble coatings could be further utilized to vary the release rate of the contaminant-reducing agents from the superabsorbent polymer matrices. These agglomerated formulations and devices could be applied to any single, joint, or multiple combination of microbial, nutrient, or film-forming agents used to control contaminants in water. Granular compositions could also be utilized in the above mentioned devices. These controlled-release compositions could also be utilized in terrestrial contaminant-controlling or reducing applications.

Generate Collection Print

L21: Entry 4 of 9 File: USPT Mar 23, 1999

DOCUMENT-IDENTIFIER: US 5885602 A

TITLE: Process for the persistence control of chemicals released into the

environment

Abstract Text (1):

The invention provides contaminant-reducing agent delivery compositions that are useful for the control of organic or inorganic contaminants in aquatic or terrestrial environments. The compositions comprise one or more superabsorbent solid organic polymers and at least one contaminant-reducing agent. These superabsorbent polymers act as the primary carriers of one or more contaminant-reducing agents effective for the control of organic or inorganic contaminants in aquatic or terrestrial environments. Contaminant-reducing agents include film-forming agents, microbial agents, nutrient agents, and mixtures thereof. The invention also provides a method to entrap and accumulate organic and inorganic contaminants in one or more superabsorbent polymer compositions containing no contaminant-reducing agent. The invention also provides a method to entrap water in superabsorbent polymers to activate natural and applied microbial and nutrient contaminant-reducing agents in terrestrial environments.

Brief Summary Text (5):

Different approaches to reducing the concentration of environmental contaminants have been explored. One such approach is bioremediation, which is the use of living organisms to break down organic and inorganic contaminants into more basic components, remove environmental pollutants, restore contaminated sites, and prevent the accumulation of further pollutants. Generally, bioremediation is accomplished using bacteria, although other microorganisms, namely fungi and algae, have been used. See, for example, Ronald M. Atlas, Chemical & Engineering News, Apr. 3, 1995, pp. 32-42. There still exists a need, however, to reduce the amount of organic and inorganic contaminants in aquatic and terrestrial environments.

Brief Summary Text (15):

The invention may be generally practiced as follows. Where a chemical additive is used for bioremediation or control, the rate of release of the chemicals from the composition of the invention may be selected to act to effectively eliminate the intentionally added beneficial chemistry after a defined period. For example, if the intended beneficial chemicals were to be added as 1,000 liters in a lake having a circumference of 4 km, the concentration of the remediation chemicals could be quantified as to their level of addition to stoichiometrically eliminate the intended chemicals when the full amount (or near full amount) of the remediation chemicals were added from the compositions of the present invention. Because the release of the remediation chemicals may be timed, the eventual elimination or conversion of the intended chemicals may be effectively timed. Where the remediation mechanism is a microbial agent, some additional effort must be done in consideration of the rate of growth or reproduction of the microbial agent if it is capable of living and reproducing outside the composition in which it is introduced into the aquatic or terrestrial environment. The control of the environment is simplified where the microbial agent must be supported in the carrier medium (the superabsorbent polymer) since there is less likelihood of uncontrolled growth of the microbial agent in the aquatic or terrestrial environment. Even if the microbial agent will thrive in the aquatic or terrestrial environment, the selection of agents which will grow at appropriate rates in the particular environment, the selection of weakened strains, and other factors within the control of the user will be selected

by the ordinarily skilled artisan to timely control the concentration and persistence of the intended chemical agent.

Brief Summary Text (16):

Surprisingly, it has been found that certain superabsorbent polymers are useful as matrices for contaminant-reducing agents effective for reducing the concentration of organic and inorganic contaminants, in aquatic or terrestrial environments, with a single application or multiple applications of a solid or liquid formulation. These compositions may be in controlled-release form.

Brief Summary Text (17):

Superabsorbent polymers, including starch graft copolymers, are well known in the art. See, for example, those described in U.S. Pat. No. 4,375,535 (Kightlinger et al.), and U.S. Pat. No. 4,497,930 (Yamasaki et al.). These polymers have been used as adhesives, flocculents, and as water-retaining materials for agricultural or sanitation uses. Superabsorbent polymers have also been used alone in pest management applications or in combination with pesticides or insecticides as matrices for their release in aquatic or terrestrial environments. However, the advantages of using this class of polymers as contaminant-entrapment matrices, as matrices for water-activation of contaminant-reducing agents, and as matrices for the controlled release of active agents in aquatic or terrestrial environments to control organic or inorganic contaminants have gone unrecognized until the present invention.

Brief Summary Text (19):

It has been observed that certain superabsorbent polymers that are impregnated with one or more contaminant-reducing agents have the ability to swell in water and/or biodegrade/degrade, and thereby release the substance(s) contained therein. Furthermore, it has been observed that the superabsorbent polymers that swell when exposed to water also have the ability to reform or contract to a consistency similar to their original form when evaporation has caused the water to be removed from the polymer matrix, and then reswell with additional exposure to water. This ability to be functional after repetitive periods of exposure to water is advantageous for pretreatment and/or prolonged controlled release applications. Certain superabsorbent polymers also have the ability to entrap and accumulate, i.e., concentrate, contaminants to facilitate their removal. Also, certain superabsorbent polymers (e.g. starch-graft) applied to terrestrial habitats can promote the degradation of contaminants by natural or applied microorganisms by entrapping water in surface or subsurface areas, and thereby provide higher and prolonged moisture levels and added nutrients (i.e., starch) for enhanced microbial growth and development. Similarly, the water entrapped within superabsorbent polymers can also activate and prolong the activity of natural or applied nutrient contaminant-reducing agents.

Brief Summary Text (35):

As used herein, the term "microbial agents" is meant to include microorganisms that enhance biodegradation processes. These microorganisms include bacteria, fungi, and algae, or combinations of these microorganisms, for example. Furthermore, as used herein, "biodegradation" means the chemical alteration and breakdown of a substance to usually smaller products caused by microorganisms and their enzymes. Contaminants that are biodegradable include any compound that can be microbially mineralized into carbon dioxide, water, ammonia and/or chloride, or that can be transformed into a non-hazardous intermediate. "Bioremediation" refers to the use of living organisms, primarily microorganisms, to convert harmful organic or inorganic contaminants into carbon dioxide, water, cell tissue, and energy, to remove pollutants from the environment, to restore contaminated sites, and to prevent further pollution from accumulating. Generally, contaminants amenable to bioremediation include: petroleum hydrocarbons, such as gasoline, diesel, fuel oil, crude oil, refinery sludges; aromatics, such as benzene, toluene, xylene, ethylbenzene, styrene; polynuclear aromatic hydrocarbons, such as naphthalene, phenanthrene, pyrene, benzo[a]pyrene; alcohols, such as isopropanol, ethanol, ethylene glycol, t-butanol; carbohydrates, animal fats and greases; detergents; ketones, such as acetone, methyl ethyl ketone; phenols, such as chlorophenol, pentachlorophenol; polychlorobiphenyls; phthalates; chlorinated solvents, such as methylene chloride, ethylene dichloride.

Brief Summary Text (48):

C. Controlled Release Contaminant-Reducing Agent Delivery Composition

Brief Summary Text (50):
In particular, the present invention is directed toward a method of formulating one or more superabsorbent polymer(s) with one or more contaminant-reducing agents, with or without water or other additives, into compositions such as solid powders, dusts, granules, agglomerates, pellets, briquets, extrusions, laminates, or composites, or into sprayable, pumpable, or injectable, variable-viscosity water or oil base gel or semi-gel like formulations that can release one or more active ingredients to simultaneously or concurrently control a variety of inorganic or organic contaminants with a single or multiple application of a solid or liquid single or multi-product formulation. Preferably, the release occurs in a controlled manner.

Brief Summary Text (51):

Impregnation of superabsorbent polymers with fatty alcohol film-forming agents such as POE-2-isostearyl alcohol or sorbitan monooleate appear to delay or slow down the rate of water absorption of superabsorbent polymers such as SUPER SORB or WATER LOCK G-100, thereby providing another useful mechanism for slow or controlled release of contaminant-reducing agents.

Brief Summary Text (52):

The slow or controlled release process could be further modified or delayed by the degree of compaction of the powdered or flaked superabsorbent polymer and superabsorbent polymer/contaminant-reducing agent formulations, by varying the size of an orifice or the number of orifices in a container into which the formulation is placed, by varying the concentration of film-forming agent, by varying the concentration of different types of superabsorbent polymers, and by adding one or more binders. For example, using one or more cationic, anionic, or nonionic surfactants or surface active agents in the composition can regulate the rate and duration of delivery (i.e., increase or decrease) of the contaminant-reducing agent from the superabsorbent polymer matrix. Additionally, a coating can be utilized to alter the swelling potential of the superabsorbent polymer, and thereby slow the rate of release of the contaminant-reducing agent from the superabsorbent polymer matrix. The coating can be hydrophobic or have varying degrees of water solubility.

Brief Summary Text (53):

It should be noted that certain salts (e.g., alkali metal halides such as NaCl) have been shown to reduce the swelling of the superabsorbent polymer matrix when introduced into water. This can have an impact on the contaminant-reducing agent delivery composition's ability to swell and control the target contaminant. Therefore, it is possible to utilize certain salts in superabsorbent polymer-based compositions as another mechanism to adjust the controlled-release rate and controlled-release profile of the contaminant-reducing agents contained therein. The rate of release will be further modified by the salinity of the environment. For example, certain superabsorbent polymers swell less in sea water, so the rate of release is slower.

Detailed Description Text (9):

A series of bioremediation bioassays were conducted in three water qualities to evaluate the efficacy of granular controlled delivery contaminant-reducing microbial compositions composed of a starch grafted sodium polyacrylate superabsorbent polymer matrix (SANWET IM-1500 LP), an acrylic acid, co-polymer release-rate regulator (PEMULEN TR-1; The B.F. Goodrich Company, Cleveland, Ohio) and a petroleum-degrading bacterial formulation (ABR Diesel Blend of viable bacterial cultures on a wheat bran base; code: 9577; Sybron Chemicals Inc., Palm Harbor, Fla.), in reducing the amount of aquatic contamination of No. 2 diesel fuel. Superabsorbent polymer granules were loaded in an aqueous formulation of bacterial and release rate regulator according to the following microsponging and entrapment procedure: 0.375 g (0.1% w/w) PEMULEN TR-1 was added to 337.12 g (89.9% w/w) water purified by reverse osmosis filtration (RO) in 500 ml NALGENE bottles and vigorously mixed on a STROKEMASTER paint shaker for approximately 30 minutes. Next, 37.5 g (10% w/w) ABR Diesel Blend was then added to the aqueous formulation and mixing was continued for an additional 60 minutes. Subsequently, 5.0 g (w/w) SANWET IM-1500 LP superabsorbent polymer granules were then added into the aqueous bacterial formulation and mixed for an additional 30

minutes to load the granules with the aqueous formulation ingredients. Swollen (hydrated) granules containing the formulation ingredients were rinsed with approximately 1000 ml RO water on a 10 mesh sieve to remove excess coating. Swollen granules were dried in a low-humidity room maintained at approximately 27-38% relative humidity (RH) and 24.degree.-26.degree. C. for 96 hours. Dry, dehydrated granules were placed into 40 ml glass vials and stored until use. Granule composition was approximately 59.0% (w/w) ABR Diesel Blend, 0.6% (w/w) PEMULEN TR-1, and 40.4% (w/w) SANWET IM-1500 LP superabsorbent polymer.

Detailed Description Text (11):

Granular controlled release SANWET IM-1500 LP superabsorbent polymer-base nutrient compositions utilized in conjunction with the bacterial composition of Example 3 consisted of a balanced blend of nitrogen, phosphorus, and micronutrients (BI-CHEM Accelerator II Special; code: 9546; Sybron Chemicals Inc., Palm Harbor, Fla.) and a release-rate regulator (PEMULEN TR-1). Superabsorbent polymer granules were loaded in an aqueous formulation of nutrients and a release-rate regulator ingredients. The following microsponging and entrapment procedure was utilized to formulate controlled delivery granules for bioassay: 0.375 g (0.1% w/w) PEMULEN TR-1 was added to 337.12 g (89.9% w/w) water purified by reverse osmosis filtration (RO) in 500 ml NALGENE bottles and vigorously mixed on a STROKEMASTER paint shaker for approximately 30 minutes. Further, 37.5 g (10% w/w) BI-CHEM Accelerator II Special was then added to the aqueous formulation and mixed for an additional 60 minutes. Next, 5 g (w/w) SANWET IM-1500 LP superabsorbent polymer granules were then added to the water-base nutrient formulation. Swollen (hydrated) granules were rinsed with approximately 1000 ml RO to remove excess coating. Swollen granules were dried in a low-humidity room at approximately 27-38% relative humidity and 24.degree.-26.degree. C. for 96 hours. Dry, dehydrated granules were placed into 40 ml glass vials for storage until use. Granule composition was approximately 62.1% (w/w) BI-CHEM Accelerator II Special, 0.6% (w/w) PEMULEN TR-1, and 37.3% (w/w) SANWET IM-1500 LP superabsorbent polymer.

Detailed Description Text (13):

A series of comparative bioremediation granule-transfer bioassays were conducted to determine the efficacy of controlled delivery superabsorbent polymer-based granular formulations composed of contaminant-degrading bacterial microorganisms and nutrients in reducing the level of petroleum contaminants in aquatic habitats having three types of water quality. The target petroleum contaminant was No. 2 diesel fuel.

Detailed Description Text (16):

Two granular application rates for each bacterial and nutrient composition were evaluated in the 3 water qualities containing the petroleum contaminant. Rates were applied to achieve bacterial levels of approximately 10.sup.5 -10.sup.6 cfu (colony forming units)/ml. A low granular application rate consisted of applying 0.35 g (approximately 292 granules at 0.0012 g/granule) of SANWET IM-1500 LP superabsorbent polymer granules loaded with ABR Diesel Blend (i.e., 0.2 g ABR Diesel bacterial cultures) and 0.65 g (approximately 342 granules at 0.0019 g/granule) of SANWET IM-1500 LP superabsorbent polymer granules loaded with BI-CHEM Accelerator II Special nutrient complex to the surface of each crystallizing dish containing 25,000 ppm No. 2 diesel fuel on RO, 50% seawater, and 100% seawater. At this application rate, controlled delivery ABR Diesel bacteria granules contained approximately 2.times.10.sup.6 cfu/ml when applied to the petroleum-contaminated aquatic habitats.

Detailed Description Text (17):

A high granular application rate consisted of applying 0.70 g (approximately 584 granules at 0.0012 g/granule) of SANWET IM-1500 LP superabsorbent polymer granules loaded with ABR Diesel Blend (i.e., 0.4 g ABR Diesel bacterial cultures) and 1.3 g (approximately 684 granules) of SANWET IM- 1500 LP superabsorbent polymers loaded with BI-CHEM Accelerator II Special nutrient complex to the surface of each crystallizing dish containing 25,000 ppm No. 2 diesel fuel on RO, 50% seawater, and 100% seawater. At this application rate, controlled delivery ABR Diesel bacteria granules contained approximately 4.times.10.sup.6 cfu/ml when applied to the petroleum-contaminated aquatic habitats.

Detailed Description Text (20):

Duration of effective controlled delivery of satisfactory concentrations of ABR Diesel Blend bacterial cultures and BI-CHEM Accelerator II Special was determined by transferring the hydrated bacteria and nutrient loaded granules to new test chambers (i.e., crystallizing dishes) containing new water and No. 2 diesel fuel, and evaluating the rate and duration of biodegradation of the diesel resulting from the release of bacteria from the granules. Comparative qualitative evaluations were based on the visual degradation (i.e., emulsification and reduction in size) of the surface layer of the No. 2 diesel fuel in all water quantities on a daily basis. Quantitative evaluations were based on diesel degradation in the high bacterial and nutrient application rate. Comparative post-treatment analyses of the concentration of diesel in tests and controls were only conducted in 100% seawater. Analyses of the average Total Petroleum Hydrocarbons (TPH) at various posttreatment intervals were conducted in pooled samples of the 3 water qualities according to EPA protocol 418.1 (Sybron Chemicals Inc.). At these posttreatment periods, the bacterial and nutrient granules were removed from each water quality, rinsed through a basket strainer, and transferred to new test chambers containing 495.0 g of RO, 50% seawater, or 100% seawater, 3 g BI-CHEM Accelerator II Special, and 5 g No. 2 Diesel fuel (i.e., 10,000 ppm). A series of granule transfers were evaluated (e.g., Transfers #1, 2, and 3).

<u>Detailed Description Text</u> (21):

Visual observations of the petroleum layer indicated a gradual emulsification/gellying and reduction in the size of the layer of No. 2 diesel fuel when compared to controls. The rate of biodegradation appeared to be slightly faster in the standards when compared to the superabsorbent polymer granular compositions; however, this was attributed to the lower level of free bacterial cultures initially present in the water column due to the slow rate of release of the bacterial from the superabsorbent polymer granules. Similar visual signs of biodegradation of the No. 2 diesel fuel <u>layer</u> were recorded in all <u>granule</u> transfer tests. Comparative observations indicated that the speed of degradation was directly related to the application rates of the loaded superabsorbent polymer <u>granules</u>, i.e., degradation of the diesel <u>layer</u> appeared to be about twice as fast in tests at the high bacterial and nutrient rates when compared to tests conducted at the lower rates.

Detailed Description Text (23):

IM-1500 LP Superabsorbent Polymer-base bacteria and nutrient granules transferred from the 25,000 ppm diesel-contaminated water qualities at 21-days posttreatment to the 10,000 ppm No. 2 diesel fuel water qualities were observed to be effective in slowly degrading the petroleum-contaminant in the 3 water qualities. Emulsification of the diesel layer resulting in a gellying and reduction in the diameter of the surface layer of diesel was observed to slowly increase over the initial 21-day test period. A second series of analyses (Transfer #1) of the diesel contamination in the 3 water qualities treated with the controlled delivery superabsorbent polymer-base bacterial and nutrient granular composition at 19 days posttransfer (i.e., 40 days posttreatment) resulted in average TPH levels of 7100, 455, and 488 mg/l in RO, 50% seawater, and 100% seawater tests, respectively. The TPH levels in the controls were 9,810-10,080 mg/l. Similar visual signs of biodegradation of the diesel layer were observed over the 19 day posttransfer period in the 3 water qualities.

<u>Detailed Description Text</u> (24):

It should be noted that visual observations of the third transfer at 14 days post-transfer of the superabsorbent polymer granules (i.e. 54 days posttreatment) containing the bacterial and nutrients to new diesel-contaminated (i.e., 10,000 ppm) water (RO, 50% seawater, and 100% seawater) indicated that the rate of biodegradation of the surface layer of diesel was comparable to the second transfer. Granules were still active when the test were terminated, suggesting that the starch-grafted superabsorbent polymer granules were still effective in releasing satisfactory levels of the diesel-degrading bacterial and nutrients for a prolonged period. In addition, it was possible that the starch-based granules also served as "mini-fermenters" for regenerating bacteria within the starch-grafted matrix for slow-release into the water. Different degrees of biodegradation of the granules were observed in the transfers.

Detailed Description Text (26):

In general, results of bioassays indicated that superabsorbent polymer granules can be used as matrices for the prolonged controlled release of bacterial and nutrients used for bioremediation of petroleum contaminants in aquatic environments having a variety of water qualities. Controlled release of the contaminant-reducing agents appeared to be via a swelling controlled diffusion process and gradual biodegradation of the superabsorbent polymer matrices. The rate of petroleum biodegradation was directly related to the application rate of bacteria and nutrient granules. Bioassay results further suggested that the dense controlled-release granular compositions would produce petroleum contaminant-reducing efficacy in shallow water, but would be required to float at or near the surface of the water to deliver the contaminant-reducing agent(s) in proximity to the surface contaminant in deep water habitats such as oceans. Results also suggested that the starch-grafted superabsorbent polymer granules may serve as a nutrient source for regeneration of the bacteria for prolonged controlled release.

Detailed Description Text (28):

A series of terrestrial bioremediation bioassays were conducted in soil to evaluate the efficacy of powdered controlled delivery admixture formulations composed of a starch-g-poly(2-propenamide-co-2-propanoic acid, sodium salt) superabsorbent polymer (WATER LOCK A-140), a No. 2 diesel fuel-biodegrading bacterial formulation (i.e., ABR Diesel Blend of viable bacterial cultures on a wheat bran base; code: 9577), and a nutrient blend (i.e., BI-CHEM Accelerator II Special; code: 9546) in reducing the levels of a petroleum contaminant on land. The target contaminant utilized in these terrestrial bioremediation bioassays was No. 2 diesel fuel (i.e., 25,000 ppm/petri dish habitat). Tests were conducted in uncontaminated field collected soil used for fill at the Lee County Mosquito Control District. Tests were replicated 3 times.

Detailed Description Text (30):

In the protocol for superabsorbent polymer-base compositions, a premixed (5 minutes) formulation of 0.07 g (0.01% w/w) ABR Diesel Blend of viable bacterial cultures on a wheat bran base (code: 9577), 12.60 g (1.15% w/w) BI-CHEM Accelerator II Special nutrient blend (code: 9546), and 12.67 g (1.15% w/w) superabsorbent polymer powder (WATER LOCK A-140) was thoroughly blended into the diesel-contaminated soil with a KITCHENAID KSM 90 mixer for approximately 7 minutes to achieve a homogeneous mixture of the components. Next, 153.62 g of this superabsorbent polymer/soil composition was added to each 100.times.15 mm glass petri dishes and lightly hand compacted to level the soil-controlled release admixture. Throughout the soil surface and subsurface, 5 ml RO water was pipetted to provide a moist soil environment and swollen superabsorbent polymer matrices of bacteria and nutrients throughout the soil. The soil was remoistened with 5 ml RO water at 3-day intervals and aerated by turning the soil with a spatula and re-leveled at 1-3 day intervals. Each petri dish in this series contained approximately 1.times.10.sup.5 cfu/g soil (approximately 0.01 g ABR Diesel bacteria cultures on a wheat bran base), 1.80 g BI-CHEM Accelerator II Special nutrient complex, 1.81 q WATER LOCK A-140 superabsorbent polymer powder, and 3.75 g (i.e., 25,000 ppm) No. 2 diesel fuel.

Detailed Description Text (33):

Bioassay room temperature and humidity ranged from 26.degree.-33.degree. C. and 74-86% relative humidity, respectively. Biodegradation of the No. 2 diesel fuel in soil with the superabsorbent polymer and non-superabsorbent polymer-base powdered compositions was evaluated on a quantitative basis. Effectiveness of the petroleum-reducing, controlled release powdered formulation of ABR Diesel Blend bacteria, BI-CHEM Accelerator II Special nutrient blend, and WATER LOCK A-140 superabsorbent polymer matrices in biodegrading the No. 2 diesel contaminated throughout the soil was based on quantitative analyses of the concentration (mg/kg) of Total Petroleum Hydrocarbons (TPH) of pooled soil subsamples at various posttreatment intervals (EPA test SW-846 9071; Sybron Chemicals Inc.). This data was compared to the TPH levels in non-superabsorbent polymer standards and controls.

Detailed Description Text (34):

Duration of effective controlled delivery of satisfactory concentrations of bacteria and nutrients from the WATER LOCK A-140 superabsorbent polymer matrices throughout the soil was determined by transferring at 21 days posttreatment (Transfer #1) one-half of each superabsorbent polymer-base and non-superabsorbent polymer-base replicate (i.e., 75 g) containing the remaining (i.e., partially biodegraded)

diesel-contaminated soil and nutrient/ABR Diesel Blend to new glass petri dishes containing an equivalent amount of clean soil (i.e., 75 g), 1.5 g (10,000 ppm) No. 2 diesel fuel, and 1.8 g nutrients (BI-CHEM Accelerator II Special). All components were blended together with a KITCHENAID KSM 90 mixer for approximately 5 minutes to assure a homogeneous mixture before being transferred into the new test chambers.

Detailed Description Text (35):

TPH analyses of soil containing No. diesel fuel (25,000 mg/kg), and a superabsorbent polymer-base (i.e., WATER LOCK A-140) ABR Diesel Blend and BI-CHEM Accelerator II Special powdered composition resulted in an average TPH level of 10,400 mg/kg at 21 days posttreatment. At this time period, approximately 58% of the No. 2 diesel fuel was biodegraded by the ABR Diesel Blend. Diesel-contaminated soil treated with the non-superabsorbent polymer-base composition (i.e., the standard) contained an average TPH level of 11,300 mg/kg, or a diesel No. 2 reduction of approximately 55%. These results indicated that the increased biodegradation efficacy observed with the superabsorbent polymer-base bacteria and nutrient compositions was due to the slower rate of evaporation that would be expected from water entrapped within the superabsorbent polymer matrices within the soil, when compared to non-encapsulated or free water within the soil. The higher degree of soil moisture was presumed to provide a more optimal environment for bacterial growth. Perhaps the starch portion of the superabsorbent polymer-base controlled delivery composition also added an additional nutrient source for increased bacterial growth.

Detailed Description Text (36):

Analyses of the <u>controlled delivery</u> superabsorbent polymer-base soil composition that was transferred at 21 days posttreatment (Transfer #1) to an admixture of nutrient-enriched soil contaminated with an additional 10,000 ppm No. 2 diesel fuel at 18 days posttransfer (i.e., at 39 days posttreatment) resulted in an average TPH concentration of 3780 mg/kg. Transfer of the non-superabsorbent polymer-base composition resulted in an average concentration of TPH of 4810 mg/kg. In general, results of comparative transfer bioassays indicated that the starch-grafted superabsorbent polymer matrices were effective in prolonging the release of entrapped petroleum-reducing bacteria and nutrients, while providing a more optimum soil moisture level and possible nutrient source for enhanced microbial activity.

Detailed Description Text (38):

A series of terrestrial bioremediation bioassays were conducted on sand to evaluate the efficacy of controlled delivery semi-viscous, liquid (i.e., water based) compositions of a starch-g-poly(2-propenamide-co-2-propanoic acid, potassium salt) superabsorbent polymer (WATER LOCK B-204) and a petroleum-degrading bacterial culture formulation (BIOTRACK DOL; code: 9690) in effectively reducing the level of surface petroleum contamination on land. The substrate utilized for this terrestrial bioassay series was TEXBLAST sand-blasting sand. The target contaminant was No. 2 diesel fuel.

<u>Detailed Description Text</u> (40):

Bioassay room temperature and humidity ranged from 26.degree.-33.degree. C. and 74-86% relative humidity, respectively. Biodegradation of the No. 2 diesel fuel on the surface of the sand with the superabsorbent polymer-base and non-superabsorbent polymer-baseformulations was evaluated on a quantitative basis. Effectiveness of the petroleum-reducing controlled delivery, variable-viscosity formulation of BIOTRACK DOL bacterial composition and WATER LOCK B-204 superabsorbent polymer matrices in biodegrading the No. 2 diesel fuel contaminated on the surface of the sand was based on analyses of the concentration Total Petroleum Hydrocarbons (TPH) of sand subsamples at 21 days post-treatment. This data was compared to the concentration of TPH in non-superabsorbent polymer-base standards and controls.

<u>Detailed Description Text</u> (41):

Duration of effective controlled delivery of satisfactory levels of petroleum-degrading bacteria from the WATER LOCK B-204 superabsorbent polymer matrices on the No. 2 diesel-contaminated soil surface was determined by transferring at 21 days posttreatment one half of each superabsorbent polymer-base and non-superabsorbent polymer-base replicate (i.e., 75 g) containing the remaining (i.e., partially biodegraded) diesel-contaminated sand and BIOTRACK DOL to the surface of the sand in new glass petri dishes containing an equivalent amount of

clean sand (i.e., 75 g) that was surface-coated with 1.25 g No. 2 diesel fuel (i.e., $10,000~\mathrm{ppm}$).

Detailed Description Text (44):

Three different superabsorbent polymers were formulated with a film-forming or bacterial contaminant-reducing agent and inert controlled release-rate regulator ingredients to evaluate the effect of superabsorbent polymer type and inert formulation components on the concentration of active contaminant-reducing agent loaded into the granules, and the profile of release of the components from the granules into an aquatic habitat. Superabsorbent polymer granules utilized in these evaluations were a potassium polyacrylate, lightly crosslinked (ARIDALL 11250), a starch grafted sodium polyacrylate (SANWET IM-1500 LP), a partial sodium salt of a crosslinked polypropenoic acid (Dow XU 40346.00 Super Absorbent Polymer), a poly (sodium acrylate) homopolymer (SS Superabsorbent Polymer), a crosslinked polyacrylamide copolymer (ALCOSORB AB3C), a crosslinked potassium/polyacrylamide copolymer (STOCKOSORB 310K), or a copolymer of acrylamide and sodium acrylate (AQUASORB PR-3005). Release-rate regulators included inert formulating materials that can retard or accelerate the rate of delivery or diffusion of the active contaminant-reducing agent(s) from the superabsorbent polymer granules, depending on the propensity of the active agents to leach from or be bound within the polymer matrices. Several nutrient formulations with no release-rate regulators were also evaluated. Non-limiting examples of release-rate regulators utilized in the admixtures included activated carbon (UCAR Carbon Co., Inc), hydrophilic silica (DEGUSSA FK 500 LS), acrylic acid copolymers (PEMULEN TR-1 or PEMULEN TR-2), sulfonated surfactants (sodium di-isopropyl naphthalene sulfonate: MORWET IP Powder; sodium mono and di-methyl naphthalene sulfonate: MORWET M Powder; or sodium alkyl aryl sulfonate: MORWET 3008 Powder), or a fatty alcohol polyethylene glycol ether (MARLIPAL 1618/25 P6000 powder). Contaminant-reducing agents included in the formulations were nutrient complexes (BI-CHEM Accelerator II Special), bacteria cultures that were unfiltered or filtered to remove large bran particles (ABR Diesel Blend; code: 9577 or ABR Gasoline Blend; code: 9350), or film-forming agents (sorbitan monooleate or a mixture of 75% sorbitan monooleate/25% 2-ethyl butanol). Microsponging and entrapment techniques were used to load the active and inert components in an aqueous medium into the superabsorbent polymers according to a 0.5, 1, or 3 hour aqueous admixing procedure (i.e., depending on the swelling potential the superabsorbent polymer granules) that is generally described in Example 4. The loading rates that were observed when superabsorbent polymer granules were admixed with an aqueous formulation of 10% contaminant-reducing agent and 1-10% inert formulation components (i.e., 1% MORWET products, 0.5% carbon and 0.1% silica for sorbitan monooleate-base compositions; or 10% MORWET or MARLIPAL products or 0.1% PEMULEN TR-1 for BI-CHEM Accelerator II nutrient compositions; or 0.1% PEMULEN TR-1 or TR-2 for ABR Gasoline Blend bacteria cultures; or 0.1% PEMULEN TR-1 for ABR Diesel Blend bacteria cultures) were as follows:

Detailed Description Text (114):

Results of formulation studies indicated that the concentration of film-forming and bacterial contaminant-reducing agents loaded into the superabsorbent polymer granules via an aqueous microsponging procedure was a function of the type of superabsorbent polymer and/or the inert release-rate regulators utilized in the admixtures. Release-rate regulators also functioned as loading-level regulators. A series of visual demonstrations were designed to evaluate the effect of the superabsorbent polymer and the release-rate regulators on the controlled release of the petroleum contaminant-reducing agents from the superabsorbent polymer granules.

Detailed Description Text (117):

Duration of controlled release of film-forming contaminant-reducing agents from floating agglomerated superabsorbent polymer-base compositions and devices was evaluated in 5 gallon plastic buckets containing 4 gallons of 100% seawater (INSTANT OCEAN) and/or in experimental fresh-water ponds (50.times.50 ft.) having a variety of natural vegetation and invertebrate and vertebrate organisms. Film-forming agents utilized in the tests were sorbitan monooleate, a mixture of 75% sorbitan monooleate/25% 2-ethyl butanol, or POE(2)isostearyl alcohol. WATER LOCK A-140 superabsorbent polymer was formulated in the agglomerated compositions used in the bioassays and field evaluations; however agglomerated compositions were also prepared using Water Lock A-100, A-120, SUPER SORB, FAVOR CA100, and AQUA KEEP

J-500.

Detailed Description Text (118):

Agglomerated compositions of WATER LOCK A-140 and film-forming agents were prepared in the following manner: 100 g sorbitan monooleate, 75 g sorbitan monooleate/25 g 2-ethyl butanol, or 100 g POE(2) isostearyl alcohol were added to 300 g acetone in a stainless steel bowl and blended with a KITCHENAID KSM 90 mixer (wire whip attachment) for 5 minutes on #2 speed. While mixing, 100 g WATER LOCK A-140 superabsorbent polymer powder was slowly added into a film-forming agent. Mixing was continued until the acetone had been driven off and the powdered composition was essentially flowable (approximately 2-3 hours). Next, 15 g of powdered superabsorbent polymer/film-forming agent composition was tightly packed into preformed, water soluble 2 mil polyvinyl alcohol bags (MONOSOL 8000 Series) and heat sealed. The encapsulated compositions were cured in a room maintained at ca 27.degree. C. and 80% relative humidity for approximately 96 hours before being transferred to a drying room maintained at approximately 27.degree. C. and 27-38% relative humidity for an addition 96 hours. 30 or 40 mesh nylon netting was then tightly or loosely wrapped around each bag to allow for different degrees of water-activated superabsorbent polymer swelling or expansion. Tightly fitted bags measured approximately 38.times.51 mm while loose fitting mesh bags were approximately 76.times.102 mm. A small cork or plastic float was attached to each end of a mesh bag to assure that the agglomerated compositions would be in contact with and just below the surface of the water in proximity to a floating contaminant. Powdered superabsorbent polymer-base film-forming agent compositions were also agglomerated into briquets or disquets by hand compaction of the compositions at various pressure in plastic tissue embedding molds (PEEL-A-WAY) or in plastic petri dishes (approximately 35.times.10 mm). 1:1 powdered controlled delivery compositions of superabsorbent polymers and ABR Diesel Blend or ABR Gasoline Blend or ABR Hydrocarbon Blend or BI-CHEM Accelerator II Special, with or without one or more release-rate regulators, were agglomerated and optionally encapsulated within water-soluble polyvinyl alcohol bags, or hand compacted into briquets or disquets as described above. Similar powdered compositions containing joint or multiple combinations of products such as 1:1:1 or 1:1:1:1 admixtures of a superabsorbent polymer, an ABR bacterial culture, a nutrient complex, and/or film-forming agent were agglomerated into a variety of matrix forms. By aligning the floating or submerged compositions in various arrangements and combinations, e.g., in layers, in tandem, linked together in a chain-type configuration, etc., one or more contaminants could be simultaneously contained and/or controlled for prolonged periods. Granular controlled-release compositions could be utilized in the same fashion. These controlled-delivery compositions would also have significant applications for the control of surface or subsurface contaminants in terrestrial habitats. Procedures for admixing, curing, drying, and hardening of the compositions are described above and in the examples.

Detailed Description Text (119):

Results of tests in fresh water ponds (approximately 240-280 umhos/cm) dusted with talc (i.e., to visualize spreading of the film-forming agents) indicated that the controlled release rate of a film-forming agent from the tight mesh bag was slower than from the loose mesh bag. Duration of film-forming agent persistence (i.e., film pressure) was monitored with ADOL (oleyl alcohol) indicator oil. Daily water temperature was approximately 35.degree. C. Mesh size also affected the rate of delivery by restricting or slowing the release of expanded superabsorbent polymer-base components into the water. Bioassay results in buckets containing 100% seawater indicated that release of the film-forming agents from the superabsorbent polymer compositions was significantly slower than observed in the fresh water due to the general inhibition of superabsorbent polymer swelling in high salinities. Similar trends in release due to mesh tightness and mesh size were also observed.

Detailed Description Text (120):

In general, these observations suggested that various devices could be fabricated that could regulate the rate of <u>delivery of the contaminant-reducing or controlling</u> agent by restricting the polymer swelling potential and/or surface area of the polymer in contact with the water, for example, by varying the size and number of openings or pores in a hollow, floating or submerged device. Further modification in release rate could be obtained by selecting superabsorbent polymers with different

swelling and biodegradation parameters and/or by admixing various release-rate regulators ingredients into the formulation. Low pressure and high pressure compaction techniques and the use of various binders and hydrophobic or insoluble coatings could be further utilized to vary the release rate of the contaminant-reducing agents from the superabsorbent polymer matrices. These agglomerated formulations and devices could be applied to any single, joint, or multiple combination of microbial, nutrient, or film-forming agents used to control contaminants in water. Granular compositions could also be utilized in the above mentioned devices. These controlled-release compositions could also be utilized in terrestrial contaminant-controlling or reducing applications.

Generate Collection Print

L21: Entry 5 of 9 File: USPT Jan 26, 1999

DOCUMENT-IDENTIFIER: US 5863789 A

TITLE: Microorganism-holding carrier and method for remediation of soil employing

the carrier

Brief Summary Text (10):
On the other hand, soil remediation utilizing a microorganism, namely bioremediation, is promising.

Brief Summary Text (11):

The bioremediation methods include strengthening of self-cleaning function of ecosystem by activating a native microorganism in the soil to decompose the polluting substance into an innoxious substance; and include also, as an improved method, intentional introduction of microorganism having the ability to decompose the polluting substance from the outside to accelerate the remediation of the polluted soil.

Brief Summary Text (17):

On the other hand, in addition to the improvement of survivability of the applied microorganism, the bioremediation involves another problem on efficiency of diffusion of the applied microorganism in the soil. The bioremediation is directed to a polluted land over a large area which contains a pollutant at a low concentration, and therefore cannot be treated by a physicochemical method such as vacuum extraction. To clean such soil practically, the decomposing microorganism is required to diffuse in the polluted soil. However, the microorganism cannot easily migrate in the soil. Therefore, a method needs to be developed for bringing the microorganism to the proximity to the polluting substance in the soil. Currently conducted methods include injection of a decomposing microorganism into soil by pressure of water or air, application of a large amount of a decomposing microorganism, and so forth. With such a method, however, the diffusion of the microorganism is extremely impeded in some kinds of soils such as soils of a high clay content or of a low water content. Therefore, an improved method is required.

Detailed Description Text (12):

Incorporation of the inducer into the carrier-constituting material is conducted by simple mixing with the carrier-constituting material, dispersion as liquid bubbles, incorporation of microcapsule made of the carrier-constituting material, impregnation into or adsorption by a porous material. In mixing the carrier-constituting material with an inducer, the rate of release of the inducer can be controlled by constituting the carrier such that the inducer-containing portions and the biodegradable or water-soluble portions are distributed mixedly.

Detailed Description Text (29):

If it is required to construct the mixture more precisely than the above, granulation is conducted so that a layer of the porous matter is formed on a part or the entire of the periphery of the highly water-absorbent polymer carrier, or the porous matter particles are embedded to the surface of the highly water-absorbent polymer carrier.

Detailed Description Text (35):

The highly water-absorbent polymer does not absorb or supplies (gradually releases) water one-sidedly, but it can repeat cycles of absorption and gradual release in accordance with the water content of the soil. Therefore, a polluting substance in a

low concentration in soil of low water content, which cannot be decomposed readily, can be decomposed acceleratedly in a following manner. A highly water-absorbent polymer holding a decomposition microorganism without water absorption is applied to polluted soil. To this soil, water or a culture medium is added in consideration of the water content of the soil. The added water or a culture medium into which a small amount of the polluting substance has come to be dissolved is gradually absorbed by the applied highly water-absorbent polymer and thereby is brought close to the decomposition microorganism. At this time, a part of the polluting substance is decomposed by the microorganism. As the water content of the soil, which had once increased by addition of the water or the culture medium, decreases with lapse of time, the absorbed water is gradually released from the highly water-absorbent polymer. In this step also, the polluting substance in the released solution is decomposed by the microorganism. After a certain time when the water content of the highly water-absorbent polymer has decreased, water or the culture medium is again added thereto. Thereby, still remaining small amount of the polluting substance is dissolved in the liquid, and the liquid is absorbed by the highly water-absorbent polymer to decompose the substance by the microorganism in the same manner. By repetition of the steps, the soil containing a polluted substance at a low content and water at a low water content can be cleaned efficiently. This method, in contrast to conventional bioremediation which collects the polluting substance diffused in the soil, is characterized in that a polluting substance is transferred in the soil by utilizing the water absorbing power as the driving force to collect the polluting substance close to the decomposition microorganism. This method makes easy the cleaning of the soil in which a microorganism does not readily diffuse, e.g., soils of a high clay content. The addition of the water or the liquid culture may be conducted in a conventional manner.

Detailed Description Text (48):

In another method for controlling the inducer diffusion rate, the diffusion barrier and/or the inducer-holder is constructed from a functional material which changes its physical, chemical, or structural properties by a factor controllable from the outside. For example, a lipid bimolecular membrane is known to increase its permeability to a low molecular compound around the phase transition temperature, and this property enables the control of gradual release of the inducer by control of the temperature. Similarly, the inducer-holder can be designed such that the gradual release of the inducer may be controlled by changing the holding capacity of the polymer as the inducer-holder by controlling various factors such as temperature, pH, salt concentration, application of electromagnetic field, and so forth. For example, by use of an N-acrylamide type polymer or the like as the polymer, the inducer diffusion rate can be controlled by an external factor such as temperature and pH. In such a manner, a bioreactor can be readily controlled by controlling an external factor to obtain the intended biological activity.

Detailed Description Text (117):

When the inducer and the inducer-holing polymer are hydrophilic, the inducer tends to diffuse and be lost through the microorganism-holding polymer which is hydrophilic. However, by interposing a diffusion barrier between the microorganism carrier and the inducer-holder, the release of the inducer can be controlled to be more gradual, thereby the inducer being supplied to the microorganism stably for a long term. With the diffusion barrier made of a functional film such as lipid film, in particular, a bimolecular lipid film, the gradual release of the inducer can be controlled by temperature, whereby the intended activity of the microorganism can readily be controlled.

Generate Collection Print

L21: Entry 8 of 9 File: USPT Aug 23, 1994

DOCUMENT-IDENTIFIER: US 5340376 A

TITLE: Controlled-release microbe nutrients and method for bioremediation

Abstract Text (1):

A <u>controlled-release</u> nutrient source is added at a low level to a <u>bioremediation</u> environment to enhance <u>microorganism</u> growth and activity and promote the effectiveness of the bioremediation in removing environmental contaminants.

Brief Summary Text (4):

Bioremediation refers to the conversion of toxic environment contaminating compounds into innocuous substances by way of microbial digestion. Bioremediation has been successfully used to treat contaminated soil in above-ground treatment systems, above-ground slurry bioreactors, slurry pits, above-ground soil heaps, composting material, and in situ. A good example of in situ soil treatment came following the Exxon Valdez oil spill in Prince William Sound, Alaska. This oil contaminated miles of Alaskan shoreline and an approximately 70 mile section of shoreline was treated using bioremediation. This remediation process as it was employed focused on enhancing the indigenous microorganisms' growth and oil degrading activities through the application of nutrients.

Brief Summary Text (5):

Representative disclosures of bioremediation process include U.S. Pat. No. 4,035,289, to Michel Guillerme et al., which discloses a method for removing hydrocarbon residues from the effluents from oil well drilling. This method involves culturing microorganisms in a portion of the effluent and then adding the portion back to the remainder to degrade the hydrocarbons. Genetic Engineering News, vol. 9, No. 10 (Nov.-Dec. 1989) at page 3 presents a good example of the in situ bioremediation processes used to assist the clean-up of the Exxon Valdez oil spill in Prince William Sound, Alaska.

Brief Summary Text (6):

It is known that speeding the bioremediation process, by promoting the growth and activity of the waste-degrading microorganisms is desirable. In most situations the microorganisms naturally present in the soil and groundwater are capable of degrading the contaminating compounds. For a successful remediation, the bioremediator must enhance the growth and activity of these naturally occurring microorganisms. To that end, it is understood that supplying the microorganisms with nutrients and advantageous environmental conditions is beneficial. Just noted U.S. Pat. No. 4,035,289 teaches the addition of nitrogen and phosphorus sources to its culturing medium. U.S. Pat. No. 4,727,031 to Richard A. Brown et al., describes a composition of nutrients and a method of using the composition to stimulate the growth of aerobic bacteria, and particularly bacteria capable of hazardous waste degradation. This patent makes reference to Busch, Aerobic Biological Treatment of Waste Waters, Oligodynamics Press, Houston (1971), at page 107, for teaching that phosphorus and nitrogen are critical growth-limiting nutrients and when not present must be added to aerobic bacteria, such as those found naturally occurring in soil and water environments. Other similar disclosures of bioremediation include U.S. Pat. No. 3,846,290 to Richard Raymond, which discloses the advantageous injection of nutrients into subsurface water supplies to reduce contaminating hydrocarbons; U.S. Pat. No. 4,401,569 to Vidyut Jhaveri et al., which similarly shows injecting nutrients into the ground to enhance microbial action on contaminants; U.S. Pat. No. 4,925,802 to Michael Nelson et al., which shows adding an amino acid to

bioremediation systems; U.S. Pat. No. 4,849,360 to Edward Azarowicz, which shows a multitank digestion process for degrading oily wastes; and U.S. Pat. No. 4,493,895 to Joseph F. Colaruotolo et al., which shows particular microorganisms which are capable of dissimilating halogenated compounds into the natural carbon cycle.

Brief Summary Text (7):

In general, therefore, bioremediations are speeded by adding nutrients, pH adjusters, and if aerobic microorganisms are used, oxygen to the soil and/or water of interest. By adjusting these parameters the indigenous microorganisms will multiply and become more active resulting in faster waste degradation. It only becomes necessary to add "foreign" microorganisms to the contaminated environment if the indigenous microorganisms do not posses the genes needed to create the enzymes necessary to degrade the contaminant, if the contaminant is at such a high concentration as to be toxic to the natural microorganisms, or if the contaminant concentration is so low the natural level of microorganisms cannot further degrade it to an acceptable level.

Brief Summary Text (8):

In most cases, bioremediations are performed in environments such as on site locations which can be classified as open systems. In the case of biodegradation in a closed environment, such as a batch bioreactor, it is sufficient to use an aqueous culture medium which completely and immediately supplies the microorganisms with the various nutrients required to increase degradation rates. These various nutrient elements are discussed in U.S. Pat. Nos. 4,035,289 and 4,727,031 which were noted above. Nutrient needs in open systems cannot be efficiently filled using the teachings of U.S. Pat. No. 4,035,289. This is because this patent shows the application of nutrient compounds which have virtually no ability to remain in the microorganisms' environment for extended periods in an open system. In this situation, it is necessary to apply these nutrients repeatedly to the open system throughout the remediation's duration.

Brief Summary Text (10):

U.S. Pat. No. 3,943,066, to Pierre Fusey, discloses a method for nutrient-waste mass association using an aqueous biodegradable emulsion of nitrogen- and phosphorus-containing substances with the hydrocarbon waste. In U.S. Pat. No. 4,460,692, to Jacques Tellier et al., a lipophilic microemulsion of an aqueous nutrient solution is applied in a layer upon the waste mass. These methods provide a way to associate essential nutrients onto the surface of the organic waste, but do not provide a controlled rate of nutrient release to the microorganisms' environment. In another, related disclosure, U.S. Pat. No. 4,401,762, also to Jacques Tellier et al., describes a process of culturing microorganisms using a microemulsion of nutrients and the use of this process in biodegradation settings.

Brief Summary Text (11):

The present invention addresses the problem of nutrient delivery to bioremediation environments by using controlled-release nutrient delivery systems engineered specifically for microorganisms. Controlled-release compositions have been used heretofore to provide nutrients to growing organisms, such as plants. See, for example, U.S. Pat. No. 4,657,576 to Johannes Lambie, which discloses a fertilizer composition for releasing nutrients to plants throughout the growing season; U.S. Pat. Nos. 3,300,293 and 3,252,786 to Andrew Bozzelli et al., which each relate to a slow-release fertilizer composition comprising a dispersion of urea-wax adduct in wax and its use fertilizing crops; U.S. Pat. No. 3,259,482 to Louis Hansen, which describes a slow-release fertilizer having a plurality of epoxy-polyester resin coatings and its use with plants; U.S. Pat. No. 3,232,739 to Steven Belak, which describes a polyurethane foam extended with free urea and the ability of the foams to supply urea fertilization to a plant throughout a long period of time; U.S. Pat. No. 3,252,786 to Andrew Bozzelli et al., which involves slow-release fertilizer compositions containing urea, wax, rosin, and optionally asphalt, and their use in fertilization processes; U.S. Pat. No. 3,475,154 to Haruhiro Kato et al., which describes resin-coated fertilizer particles and their use in garden settings; U.S. Pat. No. 4,120,685 to Silvio Varqiu et al., which describes fertilizers capable of achieving slow-release of nitrogen from urea-formaldehyde mixtures; U.S. Pat. No. 4,563,208 to Peter Backlund, which shows that fertilizer particles can be covered with a reaction product of urea and metal oxides; U.S. Pat. No. 4,210,437 to Robert Windgassen et al., which shows liquid fertilizer compositions which provide sulfur, nitrogen and micronutrient metals; and U.S. Pat. No. 4,756,738 to William J. Detroit, which shows a copolymer matrix which is capable of gradually releasing fertilizer.

Brief Summary Text (13):

The current invention improves the efficiency of <u>bioremediations</u> in soil and/or aqueous environments by providing <u>controlled-release</u> compositions which supply nutrients to the <u>microorganisms</u> in a regulated, environmentally sound, and cost effective manner.

Brief Summary Text (15):

An improvement in bioremediation processes has now been found. Such bioremediation processes are those in which an organic chemical-contaminated soil or aqueous environment is remediated by the digestive action of microorganisms, on the chemical contaminant, within the environment. This digestive action typically takes two or more months before acceptable levels of degradation have taken place and the contaminated environment is no longer considered a threat. The improvement provided by the present invention involves applying to said environment, and thus the degrading microorganisms present therein, a controlled-release source of microorganism nutrients at a low level and optionally vitamins and/or nutrients which double as buffering agents (to keep the environment surrounding the product at a pH which is compatible with the growth and activity of the desired microorganisms). This controlled-release source is capable of continuously supplying an effective level of microorganism-promoting nutrients, some of which may double as buffering agents, to the contaminated environment during the prolonged period of digestive action. These prolonged periods of bioremediation action are those typically associated with this process in the art, such as at least about 1 week and more commonly from 1 to 100 weeks, especially from 10 to 40 weeks. The controlled-release source of nutrients typically releases nitrogen and/or phosphorus and/or sulfur to the microorganisms. This invention can find application in bioremediations performed in soils and/or aqueous environments which have become contaminated with aliphatic hydrocarbons. It can also find use in soils and/or aqueous environments which have been contaminated with aromatic hydrocarbons, including halogenated aromatic, polynuclear aromatic, polychlorinated biphenyls (PCB), trichloroethylene (TCE), percholorethylene, various pesticides, various herbicides, and with any chemical deemed to be bioremediable.

Brief Summary Text (17):

In addition to the macronutrients (nitrogen, phosphorus, and sulfur), the invention can serve to deliver essential micronutrients such as micronutrient metals to the microorganisms as well. In another embodiment additional growth promoters, a vitamin source, for example yeast extract, can be incorporated into the controlled-release composition or administered concomitantly to additionally promote the vitality and growth of the microorganisms. Thus, in additional aspects, this invention can deliver in a controlled-sustained manner macronutrients, micronutrients, buffers, vitamins and the like or any combination thereof, to the bioremediation microorganisms.

Brief Summary Text (19):

As will be apparent, the present invention is highly advantageous when applied to environments requiring bioremediation activities in that it provides a way to efficiently deliver essential nutrients, in an environmentally sound way, to the waste-degrading microorganisms throughout the entire remediation without the expensive, wasteful, labor intensive multiple reapplications called for in methods of the prior art. Surprisingly, in order to obtain the most efficient bioremediation, the concentration of the nutrients in the soil is maintained at a lower level than suggested by the prior art.

Detailed Description Text (3):

The materials employed in the present invention deliver nitrogen, phosphorus, and/or sulfur as macronutrients to the bioremediation microorganisms. Nitrogen can be delivered as nitrate, ammonium, urea or cyanamide. Phosphorus is typically delivered as phosphate. Sulfur is generally delivered as sulfate. Potassium, a usual component of plant nutrient material, may be present but generally is not required. The

macronutrients employed to make up the controlled-release product are normally standard fertilizer materials and can include urea, ammonium nitrate, ammonium phosphates, ammonium sulfate, calcium nitrate, calcium cyanamide, sodium nitrate, calcium phosphates, single superphosphate, triple superphosphate, potassium nitrate, and potassium sulfate. These materials are highly water soluble with the exception of calcium cyanamide and calcium phosphates, and if employed without a controlled-release coating, would rapidly dissolve in and be dissipated in an open environment. The amounts of nitrogen, phosphorus, and sulfur can be varied in the product to give compositions having from 0 to 40 percent nitrogen, from 0 to 40 percent phosphorus, and from 0 to 20 percent sulfur by weight based on the overall micronutrient composition. Preferred ranges are from 10 to 40 percent nitrogen and 2 to 20 percent phosphorous and 0 to 10 percent by weight sulphur. In one preferred embodiment, the total nitrogen is approximately 28 percent nitrogen, and the phosphorous level is 3.5 percent or 8 percent in P.sub.2 O.sub.5 form. This type of product would be referred to as a 28-8-0 (0 being potassium in K.sub.2 O form) product in typical fertilizer nomenclature. At this time, it is accepted in the bioremediation industry that the product should release 1 to 10 parts nitrogen per part by weight phosphorus.

Detailed Description Text (7):

Since the microorganisms also utilize vitamins such as thiamine, folic acid, biotin, nicotinic acid, and the like, these can also be included into the controlled-release products. It has been shown that part per million concentrations, and in some cases a fraction of a part per million, of vitamins can drastically increase activities of the microorganisms and thereby increase speed of waste degradation. For this reason, from 0.01 to 100 ppm by weight of such vitamins may be included in the product.

Detailed Description Text (8):

Microorganisms also thrive best in particular pH ranges, such as from about 6.5 to 8, so a buffering system may also be added to the product to maintain the environment around the controlled-release particle at these desirable pHs. This buffering system can be made up of compounds which double as a nutrient source making the system even more desirable. For example, the phosphate phosphorous sources can provide buffering capacity into these desired pH ranges. Representative buffers are a 1:1 mixture of KOH and KH.sub.2 PO.sub.4 and a 2:1 disodium phosphate and monopotassium phosphate mixture.

Detailed Description Text (9): Controlled-Release Coating

Detailed Description Text (10):

The controlled-release compositions of this invention are particulate solid materials which comprise a water soluble core of the nitrogen and/or phosphorus and/or sulfur micronutrient source and optionally micronutrients and/or vitamins and/or buffers surrounded by a release rate-controlling coating. While any of the release rate-controlling membrane materials of the art, such as those included in the above-referenced Theeuwes or Baker patents can be used (these patents are incorporated herein by reference), it has been found in our work to be advantageous to use a resin formed from linseed oil and dicyclopentadiene (DCPD) copolymerized and bodied. Such a composition is employed commercially under the trademark "Osmocote", a trademark of Grace-Sierra Horticultural Products Company. This material is described in U.S. Pat. No. 3,223,518 and Dutch Patent No. 132,686, both of which are incorporated herein by reference. This "Osmocote" coating is advantageous due to its low cost and high effectiveness when used to create controlled-release products capable of lasting from one week to 100 weeks. A typical remediation will last from 10 to 40 weeks.

Detailed Description Text (12):

Other types of <u>controlled-release</u> coatings which can achieve the desired <u>controlled nutrient release</u> to bioremediation environments include polyethylene, polypropylene, ethylene, propylene copolymer, ethylene-vinyl acetate copolymer, vinylidene chloride, vinyl chloride, vinylidene chloride-vinyl chloride copolymer, and polystyrene. These materials are discussed in more detail in U.S. Pat. No. 4,369,055 which is incorporated by reference.

Detailed Description Text (13): Controlled-Release Nutrients

Detailed Description Text (14):

The macronutrients employed in the present process are typically granular materials having grain sizes from about 0.1 to about 5 mm. These granules will be composed of any macronutrient, micronutrient, vitamin, and/or buffer previously mentioned or amenable to being granulated and will be coated with a resin layer. This coating will most likely be the linseed oil/DCPD copolymerized resin and will be applied to the granules and then heat cured onto the granules' surface creating a controlled-release film. The amount of this resin applied to the granules will range from 1 to 20 percent by weight (based on the weight of macronutrients) depending on the granules' shape and the required longevity of the product. The typical percent of resin used will be between 5 and 15 percent.

Detailed Description Text (16):

In accord with the process of this invention, the precise amount used will depend on the level of contamination being treated, the concentration of background nutrients, the percentages of nutrients in the controlled-release product of interest, and other conditions affecting the nutrient release.

Detailed Description Text (17):

Typical use levels for soil treatment will run from about 0.50 to about 50 pounds of controlled-release nutrients per cubic yard of contaminated soil. We have found, quite unexpectedly, that even though the release of nutrient is controlled and delayed, we get best results at lower overall use levels than we observed for non-controlled release (i.e., wholly soluble) materials. More specifically, while soluble materials give best results at about 5 or 10 pounds per cubic yard, so as to obtain a contaminant carbon:nitrogen:phosphorus atomic ratio of 100:20:1, we have obtained far better results using as little as 1 pound of controlled-release material per cubic yard of soil. On this basis we prefer to use 0.25 to 5 pounds per cubic yard and especially 0.5 to 3 pounds per cubic yard.

Detailed Description Text (18):

In soil environments, moisture must be present to allow the microorganisms to flourish. Moisture may be added if desired. The <u>controlled-release</u> product may be spread on the contamination and/or tilled into the contaminated soil. In the case of contaminated aqueous environments, use levels of from about 0.0005 to about 0.5 pounds per gallon are advantageous, again this is dependant upon the environmental conditions.

Detailed Description Text (21):

The <u>bioremediation</u> processes of this invention may rely upon the microflora present in situ at the contamination site and this is typically preferred. Alternatively <u>microorganisms</u> may be seeded into the contamination site and these added materials can include any of the organisms known in the art to affect contaminant degradation such as the Pseudonomids, Methylotrophic <u>bacteria</u>, Acinetobacter as well as a variety of anaerobic <u>bacteria</u>. It is not intended that his invention be limited to any particular type or family of organisms, including genetically engineered microorganisms.

Detailed Description Text (25):

A nitrogen and phosphorus nutrient granule in controlled-release form was prepared as follows. Granules each containing ammonium nitrate, 80%; ammonium phosphates, 12%; calcium phosphates, 5%; and inerts, 3% were screened to a Tyler mesh range of -6 to +12. This material was then coated with 7.4% by weight (basis finished product) of a linseed oil/DCPD copolymerized resin (6% w linseed oil/38% w DCPD). The coating was accomplished by heating the screened nutrient granules to approximately 70.degree. C., then applying the resin at a flow rate which gives a uniform coating. This controlled-release material had a nitrogen content of approximately 28% and a phosphorus content of approximately 3.5%. The release characteristics of this product, as found in the laboratory, showed that the nutrients were released over a three month period at 70.degree. F. The lab test consisted of statistically splitting 8 grams of the controlled-release product out of the bulk and placing it in a funnel containing approximately 200 ml of washed

sand. The funnels were leached every seven days with water and the leachate analyzed for nutrients.

Detailed <u>Description Text</u> (26):

This material was applied to soil contaminated with diesel oil. It was predicted, using the old methodology of applying soluble nutrients, that the bioremediation would take four months. The contaminated soil was remediated in slightly over two months using this controlled-release product. This material was also applied to the beaches in Prince William Sound, Alaska, contaminated with oil from the Exxon Valdez, and supplied nutrients continuously to the indigenous microorganisms for approximately three months.

Detailed Description Text (28):

The preparation of Example 1 was repeated with the following changes. After applying the controlled-release resin, a dispersion containing micronutrients, vitamins, and a buffer was applied to the granules and cured. The cured dispersion consisted of 20% resin by weight with the remaining 80% consisting of phosphate buffer, magnesium, iron, potassium, manganese, molybdenum, thiamine, riboflavin, nicotinic acid, pantothenic acid, folic acid, biotin, choline, inositol, and protein. This dispersion was then overcoated with 0.5% (by weight of finished product) linseed oil/DCPD resin. Also coated with this dispersion and then overcoated was a urea substrate consisting of 25% urea granules coated with 11.1% resin and the remaining 75% urea coated with 13.8% resin. This added urea was then mixed with the micronutrient containing particles to comprise 20% by weight of total product and created a product containing approximately 29% nitrogen, and 3% phosphorus as well as micronutrients.

Detailed Description Text (29):

This product was tested in a laboratory microcosm competing against a dry soluble nutrient of comparable composition. At four weeks, the bacteria population in the controlled-release nutrient treated microcosm was an order of magnitude higher than the soluble nutrient treated microcosm and two orders of magnitude higher than the microcosm having no nutrient treatment.

Detailed Description Text (35):

Nitrogen and phosphorus nutrient granules, in controlled-release form, are prepared as follows. Granules containing urea are screened to a Tyler mesh range of -6 to +12. Granules containing calcium phosphate, monobasic are also screened to a Tyler mesh range of -6 to +12. 22.6 pounds of the urea granules are heated to approximately 65 C. and 3.4 percent linseed oil/DCPD resin is applied. Then 7.8 more pounds of screened, uncoated urea is mixed into the 22.6 pounds of partially coated urea and the mix is brought up to approximately 65.degree. C. After achieving this temperature, 6.1 percent linseed oil/DCPD resin is added by weight of substrate and resin. To conclude the coat 14.2 pounds of calcium phosphate, monobasic acid is added to the partially coated urea fractions and the temperature is brought up to approximately 70.degree. C. Resin is applied at 5.7 percent on the total weight of the coated product. The final product contains 52.5 percent urea coated with 13.8 percent resin, 17.5 percent urea coated with 11.1 percent resin, and 30.0 percent calcium phosphate, monobasic coated with 5.7 percent resin. This product will supply nitrogen and phosphorus continuously, in a ration of 5 to 1 respectively, for three months in a moist environment kept at 20.degree. C.

Detailed Description Text (37):

A bioremediation field trial was conducted to determine if <u>controlled-release</u> nutrients would enhance the degradation of diesel when compared to the standard practice of applying dry soluble nutrients. The results of the trial are presented in FIGS. 1-6.

Detailed Description Text (38):

The two controlled-release products were added at concentrations of 1, 5, and 20 lb/cu yard of soil. The first controlled-release product used is referred to as Customblen.TM. 24-89 and contains 27.5% nitrogen and 8% phosphorus in P.sub.2 O.sub.5 form. The second controlled-release product used is referred to as Max Bac.TM. 2 and contains 26% nitrogen, 10.5% phosphorus in P.sub.2 O.sub.5 format, and 0.65% potassium in K.sub.2 O form. The controlled-release products were prepared as

described in Example 1 and Example 2 with the modification that the dispersion was coated onto the substrate before the controlled-release membrane was applied.

Detailed Description Text (43):

As shown in FIG. 1, it was observed that the total bacteria present in the samples remained relatively constant among all of the samples with the exception that the high use levels (20 lb/cu yard--series E and H) of controlled release products gave lower bacterial levels than the 1 lb/cu yard or 5 lb/cu yard series (series C, D and F, G). Similarly only modest differences were noted in colony-forming unit concentration but with better performance being seen with low controlled release nutrient levels than at high levels. There were major differences in levels of phenanthrene degrading bacteria among the 8 tests. The control with no added nutrient was as much as two orders of magnitude lower than the tests with nutrient. At high use levels (20 lb/cu yard--series E and H), the added fertilizer first boosted and then depressed the number of organisms observed. At low levels (1 and 5 lb/cu yard--series C, D, G and H) the number of such organisms elevated over time by as much as 21/2 orders of magnitudes and in several samples as much as one order of magnitude greater than the standard practice addition of soluble fertilizer. In virtually all cases where nutrient was added, including the control, the level of fluorescent Pseudonomids went up dramatically in the first month and then fell in the second month.

Detailed Description Text (45):

The standard practice of adding dry soluble fertilizer reduced the contamination to about 750 ppm in 100 days. Interestingly, high loadings (20 lb/cu yard) of controlled release material gave poor results. At this condition, over 1000 ppm of contamination remained after 100 days. At a rate of 5 lb/cu yard, controlled release material reduced contamination to about 250 ppm in about 100 days. Best results were observed at the lowest rate of controlled release material addition--1 lb/cu yard. At this condition, the contamination was consumed the fastest and to the lowest level, 250 ppm.

Detailed Description Text (46):

Dirt samples initially containing added diesel contamination as noted above were bioremediated under 3 comparative conditions—condition A—no fertilizer, condition B—3.5 lb/cu yard of dry soluble fertilizer and condition F controlled release fertilizer at 1 lb/cu yard. Samples were taken at day 1, day 31, day 53, and day 95 and analyzed by gas chromatography. The results were digitized to give relative levels of each carbon number fraction. FIGS. 3, 4, 5 and 6 show the results of these analysis and illustrate a regular and striking advantage to the controlled release material which allowed the clean up to achieve a reduction in contamination to 16 units while the control was at 55 units and the soluble fertilizer test gave 41 units.

CLAIMS:

- 1. In a biological remediation process wherein microorganisms are employed to degrade contaminating organic compounds present within an environment over an extended period of time, the improvement which comprises applying to said environment a low-level of controlled-release source of microorganisms nutrients capable of continuously supplying an effective microorganism growth-and activity-promoting level of nutrients to the microorganisms over a period of time of at least about two months, said controlled-release source of microorganism nutrients being in the form of coated solid particles each having a core of water soluble microorganism nutrients encapsulated in a release rate-controlling coating and where said particles are admixed in the environment at a level of 0.25 to 3 pounds per cubic yard of environment.
- 18. The process of claim 13 wherein said <u>controlled-release</u> nutrients are comprised of water soluble macronutrients encapsulated in a linseed oil/dicyclopentadiene resin.
- 19. In a biological remediation process wherein microorganisms are employed to degrade contaminating organic compounds present within a soil environment over an extended period of time, the improvement which comprises admixing with said soil

environment from about 0.25 to 3 pounds per cubic yard of soil environment of controlled-release source of microorganisms nutrients capable of continuously supplying an effective microorganism growth-and activity-promoting level of nutrients to the microorganisms over a period of time of at least about two months.

WEST

End of Result Set

Generate Collection Print

L21: Entry 9 of 9 File: USPT Nov 2, 1993

DOCUMENT-IDENTIFIER: US 5258303 A

TITLE: Bioremediation system and method

Abstract Text (1):

An apparatus and method to clean chlorinated hydrocarbon contaminated soils with a bioremediation system is provided. Treatment beds about one-half acre in size are provided with a gravel base on an impervious film layer. A woven polyethylene membrane is laid on the gravel base with a sand layer in between the membrane and the soil layer to be treated. An aqueous treating liquid containing selective microorganisms is sprinkled on the top surface of the soil layer. Exit gases from the bioreactor are released at zero pressure through perforated pipes laid in the base aggregate layer.

Brief Summary Text (4):

Bioremediation using microorganisms has offered the most effective method of cleaning up hazardous waste sites. The use of microorganisms which are designed to break down certain chemical compounds and more importantly to selectively secrete enzymes to break down the more completely chlorinated hydrocarbons is well documented. These compounds, referred to herein as biodegradable chemical compounds, include not only partially halogenated but also fully halogenated carbon based compounds. These compounds are classified as "BOD" (biological oxidizable demand) which require additional oxygen during bioremediation with the treatment being enhanced by micronutrients, trace metals, organic acids, alcohols and other compounds. The more difficult compounds, such as PCB's, are classified "COD" (chemical oxidizable demand) requiring prior chemical reaction before digestion is effective by the microorganisms.

Brief Summary Text (11):

This invention also involves and contemplates its use not only at a site where contaminated soil is brought to be cleaned, but also in a waste treatment unit where a carrier is purposefully "contaminated" with the chemical to be ingested by the microorganisms. Thus, while the specific embodiment described treats many acres of soil, a smaller facility located directly on the grounds where small quantities of chlorinated by-products are produced is also contemplated. In this latter instance, the small quantity of chlorinated hydrocarbons may be subjected to bioremediation using the device and system of the present invention at the site where the contaminants are produced. In this configuration, the carrier is purposefully contaminated and spread on a small version of the treatment cells described hereinbelow. Further, since the bioreactor means used in the present invention can be portable, a chemical company can move this bioreactor means from plant site to plant site to treat accumulated hazardous chemicals.

Brief <u>Summary Text</u> (12):

This invention is intended to be used with a variety of strains of <u>bacteria</u> specially designed or chosen for <u>bioremediation</u> techniques described and listed in the patents incorporated by reference hereinabove as well as U.S. Pat. Nos. 3,846,290; 4,385,121; and 4,765,902, also incorporated by reference herein. The strains of <u>bacteria</u> for any particular waste compounds are well known and all may be used in the present invention including the ones listed in the references listed above.

Brief Summary Text (13):

The system and methods of this invention also include a bioreactor also described fully in the prior art. The bioreactor is a vessel packed with materials to extend the surface area on which the microorganisms can grow such as column packing materials including burrel saddles, corrugated plastic trays, and the like. The bioreactor is intended to produce huge <u>bacteria</u> counts which slough off into the aqueous medium to be transported to the site of the <u>bioremediation</u>. The bioreactor also approaches infinite contact of the aqueous medium with the <u>bacteria</u> cultures to complete bioremediation of the input liquid and gases.

Brief Summary Text (22):

Yet another aspect of the invention is a system to clean soil contaminated with biodegradable chemical compounds, wherein the soil contaminated with biodegradable chemical compounds is disposed with an exposed surface on top of a layered bed. The system includes a layered bed that includes impervious retaining means at the bottom of the bed to prevent any liquid from escaping from the bed, a base layer disposed on the impervious retaining means includes stone granules, membrane means disposed on the base means to prevent fine particle size material from entering the base layer, and separation layer means disposed on the membrane means to provide a porous layer, to aid in liquid dispersion between the layers, and to separate the carrier contaminated with biodegradable chemical compounds from the membrane means. The system further includes bioreactor means to hold and propagate a supply of microorganism means includes microorganisms to break down and digest the biodegradable chemical compound contaminants and to bring these microorganism means into intimate contact with liquid and gaseous biodegradable chemical compound contaminants. The bioreactor means further includes a vapor vent to issue vapors and a liquid outlet means to issue a treating liquid includes microorganisms. The system also includes a multiplicity of parallel aligned first perforated tubing laid in the base layer, and pump means to draw a vacuum on the first perforated tubing and transport fluid from the base layer to the bioreactor means. The system further includes first feed line means fluid flow connecting the first perforated tubing to pump means to the bioreactor means, second feed line means fluid flow connecting the vapor vent with second perforated tubing laid parallel with the first perforated tubing in the base layer, and third feed line means fluid flow connecting the liquid outlet means with a sprinkling means to spread the treating liquid over the exposed surface of the soil. It is preferred that the system include the second length of perforated tubing being of sufficient length and of sufficient porosity to reduce the pressure of the vent vapors to zero.

Detailed Description Text (6):

The system may be described as a closed vent in that there is no direct venting from the bioreactor but with a controlled release into the treatment bed system allowing the vapors to perk upwardly through the treating liquid and emerge as cleansed only carbon dioxide, oxygen and nitrogen. Thus, this system allows biotreatment of any possible vapor contaminants issuing from the bioreactor means.

Detailed Description Text (8):

Using a modified systems as described above, soils contaminated with fully halogenated hydrocarbons, such as PCB's can ultimately be treated using the aerobic digestion system described. Prior art describes bioremediation of fully chlorinated hydrocarbons utilizing an anaerobic bacteria that first attacks the chlorine atoms by forming enzymes that attach to these chlorine atoms. Once some of the chlorine atoms have been removed from the molecule, the more standard aerobic digestion techniques can be utilized. In order to handle fully halogenated compounds contaminating the soil, the above device and method must be modified. An additional unit must be included in the bioreactor means to handle the anaerobic bacteria, providing a continuous supply of these specialized bacteria with appropriate nutrients upon demand. The treatment bed is then flooded with liquid and no gases are introduced through the vent lines, other than nitrogen to purge out any oxygen entrained in the soil. The specialized anaerobic bacteria are introduced through the vent piping or vacuum piping under the liquid level and allowed to dissipate in the flooded bed. After an appropriate period of enzymatic reaction wherein some of the chlorine atoms have been removed from a major portion of the molecules of the contaminating chemicals, the system is converted to the aerobic system described hereinabove with the chemicals being treated using aerobic micro-organisms to finish the digestion and cleaning of the soil. As the chemicals are partially digested and partially broken down, the materials tend to be self-lubricating to aid in the solubilization of the compounds in the liquid phase to be drawn off by the vacuum line into the bioreactor means.

CLAIMS:

- 6. A system to clean soil contaminated with biodegradable chemical compounds comprising:
- (a) a layered bed means to hold soil contaminated with biodegradable chemical compounds disposed with an atmospherically exposed surface on top of a layered bed the layered bed comprising:
- (i) impervious retaining means at the bottom of the bed to prevent any liquid from escaping from the bed,
- (ii) a base <u>layer</u> disposed on the impervious retaining means comprising stone granules,
- (iii) membrane means disposed on the base means to prevent fine particle size material from entering the base layer, and
- (iv) separation layer means disposed on the membrane means to provide a porous layer, to aid in liquid dispersion between the layers, and to separate the soil contaminated with biodegradable chemical compounds from the membrane means,
- (b) bioreactor means to hold and propagate a supply of microorganism means comprising microorganisms to break down and digest the biodegradable chemical compound contaminants and to bring these microorganism means into intimate contact with liquid and gaseous biodegradable chemical compound contaminants, the bioreactor means comprising a vapor vent to issue vapors and a liquid outlet means to issue a treating liquid comprising microorganisms,
- (c) a multiplicity of parallel aligned first perforated tubing laid in the base layer,
- (d) pump means to draw a vacuum on the first perforated tubing and transport fluid from the base layer to the bioreactor means,
- (e) first feed line means fluid flow connecting the first perforated tubing to pump means to the bioreactor means,
- (f) second feed line means fluid flow connecting the vapor vent with second perforated tubing laid parallel with the first perforated tubing in the base layer, wherein the second length of perforated tubing is of sufficient length and of sufficient porosity to reduce the pressure of the vent vapors to zero, and
- (g) third feed line means fluid flow connecting the liquid outlet means with a sprinkling means to spread the treating liquid over the exposed surface of the soil.